CEREAL CHEMISTRY

Vol. VII May, 1930 No. 3

SOME COMPARISONS BETWEEN COMMERCIALLY AND EXPERIMENTALLY MILLED FLOURS[1]

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(Received for publication December 15, 1929)

INTRODUCTION

Wheat flour, as a finished product, ready for the baker, is the result of a long succession of processes—biological, mechanical, and chemical—all of which admit the play of almost innumerable variables. It has been the aim of the investigator interested in flour problems to control, counteract, take advantage of, or evaluate the effects of many of these variables, working toward the production of a suitable product for the making of bread.

Bailey (1925) stated that, "Properties of flour must be considered in their relation to: (first) the raw material from which it is manufactured, or wheat; (second) the process of manufacture, or milling; (third) its adaptability to the principal use to which flour is put, or baking." The first is undoubtedly the most important consideration at this time, and the plant breeder, the agronomist, and the plant pathologist are devoting their energies to the problem of improving the quality of the wheat that is grown, while the grain inspectors and the wheat blenders, as well as the cereal chemists, are devoting their efforts to evaluating the wheat which is marketed, so as to produce the highest possible quality of flour.

The adaptability of flour to baking has been subjected most thoroughly to laboratory investigation. The research along this line has been in the direction of finding the shortest, simplest, and most reliable method of evaluating the baking properties of flour without recourse to the rather lengthy and complicated baking test.

[1] Published with the approval of the Director, as Paper No. 935, Journal Series, Minnesota Agricultural Experiment Station. Condensed from a thesis presented by Truman A. Pascoe to the Graduate School of the University of Minnesota in partial fulfillment of the requirements for the Degree of Doctor of Philosophy, June, 1928.

It is well known to the miller that variations in the milling procedure will produce variations in the properties of the flour, but few quantitative data are available as to the exact nature of these variations.

The plant breeder, in an effort to improve the milling qualities of wheat through selection and breeding practices, can obtain final results in terms of baked bread only through the production of samples of sufficient size to mill into flour. The use of the laboratory mill has greatly reduced the size of sample necessary to produce a workable quantity of flour. Of necessity the milling procedure with a small mill must be quite different from that of the commercial mill.

Classification of the middlings is not attempted to the same degree as in commercial milling. No purification of these middlings is ordinarily provided. It is also probable that the control of pressures on the rolls in the reduction of middlings is less precise and liable to greater variations. A tendency to bolt the ground middlings more extensively is also frequently observed in the ordinary operation of the laboratory mill.

The question arises at once as to the effect of this modified milling treatment on the flour produced, and the advisability of using laboratory data based upon studies of such flour to prognosticate the properties of flour commercially milled from the same wheat. The same reasoning applies to the grain merchant's and miller's practice of milling samples from large shipments to gain advance information concerning the desirability of the wheat in question.

The present investigation was undertaken with these considerations in mind.

Historical

In view of the very comprehensive review of the literature on flour and its properties as related to wheat, milling, and baking, recently compiled by Bailey (1925) and the reviews of the papers on more specialized topics by other authors, no attempt will be made to present here a complete historical review of the subject. Sharp and Gortner (1923) thoroughly reviewed the literature dealing with flour strength. Rumsey (1922) discussed the historical aspects of enzyme phenomena in relation to baking strength.

The following discussion includes only those papers having a direct bearing on the problem at hand.

The Experimental Mill.—The laboratory size of roller flour mill was used by Hays and Boss (1899) in connection with wheat variety and breeding studies. This was only fifteen years after the beginning of the new era in roller milling (Edgar, 1912), and early in the present period of researches on flour.

Total Protein.—The use of the total crude protein as a measure of flour strength has been the subject of considerable controversy. Thomas (1917), in a study of the comparison of total protein content with loaf volume, showed that "high crude-protein content as a rule is accompanied by high strength, but the relation between these two factors varies with the different classes of wheat and extremely high crude-protein content is sometimes accompanied by a decrease in baking strength." Zinn (1923) calculated coefficients of correlation between loaf volume and protein content of both wheat and flour. Values of $r = +0.1827 \pm 0.0459$ were obtained for Minnesota commercial wheat varieties; $r = +0.4621 \pm 0.1011$ for Montana spring wheat varieties. Total protein of flours from Minnesota commercial wheats and from pure-strain wheats correlated with loaf volume gave coefficients of $r = +0.2586 \pm 0.0442$ and $r = +0.5469 \pm 0.0689$, respectively.

Mangels and Sanderson (1925) studied three crops of North Dakota wheats, obtaining in all cases significant positive correlations between loaf volume and total protein content of the wheat.

Nebraska flours reported by Blish and Sandstedt (1925) showed a correlation coefficient of $r = +0.304 \pm 0.058$ between loaf volume and protein content of the wheat.

Mangels (1926) computed coefficients of correlation between loaf volume and protein content of wheats for several individual years and obtained positive correlations in eight out of eleven cases.

Bailey (1924), and Bailey and Sherwood (1926) reported significant positive correlations from a large number of comparisons of protein content of straight grade flours and the loaf volume of loaves baked therefrom. In the latter paper the increase in loaf volume per increment of protein, at various protein levels, was computed.

Grewe and Bailey (1927), in comparing protein content of both hard and soft wheat flours with loaf volume, obtained the coefficient of $r = +0.678 \pm 0.091$.

Diastatic Activity.—The term diastase as applied to the starch-splitting enzyme of malt was first proposed by Payen and Persoz

(1833). Wood (1907) was apparently the first to recognize the importance of the sugar formed from starch hydrolysis in the nutrition of the yeast in a fermenting dough.

Rumsey (1922) after developing a reliable method for the quantitative determination of diastatic or saccharogenic (Bailey, 1925) action of wheat flour, investigated the relation of this property to baking strength. It was found that flours from various sources differed markedly with respect to their starch-splitting power, and other factors being equal, the differences were always reflected in loaf volume. In the fourteen flours examined by Rumsey, variations of 900% in diastatic power were encountered. The influence of time, temperature, and H-ion concentration on the activity of diastase was also studied and the optimum conditions defined. Olsen and Fine (1924) have recently clarified the question of the temperature and H-ion relationship in determining optimum conditions for diastatic action. The optimum pH for the action of malt diastase was found to change from pH 4.3 to pH 6.0 when the temperature was raised from 25° to 69°C. This is in good agreement with the earlier work of Rumsey who found at 27°C. that pH 4.7-5.0 was optimum for the action of flour diastase.

Sherwood and Bailey (1926) determined the effects of adding sprouted wheat to normal wheat to increase the saccharogenic properties of the flour milled therefrom. When two per cent of sprouted kernels were added to wheat which yielded flour having an initial diastatic value of 126 Rumsey units, the diastatic value increased to 204 Rumsey units, the loaf volume was increased 5% and other properties of the bread were improved. Sherwood and Bailey defended Rumsey's method against the criticism of Sørensen, contending that it, after all, most nearly simulates actual dough fermentation conditions.

Sørensen (1924) made the criticism that Rumsey's method did not compare different flours at the same H-ion concentration. It was suggested that after a buffer curve had been determined for a given flour, the diastatic activity should be determined at three different pH values. In this way a curve could be constructed which would be more or less characteristic for the flour and directly comparable with similar curves for other flours.

Following this suggestion, Bailey and Grewe (1927a) determined the diastatic activity of seventeen flours at the natural H-ion concentration of the flour and water mixtures, and at pH 5.0. The rather surprising results showed that those flours with highest

initial diastatic activity increased at a greater rate with changing H-ion concentration than did those with a low initial saccharogenic activity.

Sherwood (1926) reported that the diastatic activity of wheat flour is influenced by grinding. Middlings stocks and flours were tested before and after grinding. Part of the third reduction stock which passed through a 50 GG and over a 72 GG showed a diastatic power (Rumsey's method) of 53 units before regrinding and 103 units after regrinding. Another portion of the third reduction stock which passed through a 50 and a 72 GG, but over a 10 XX, showed 40 units before and 73 units after regrinding. The stocks which tailed off the 10 XX in the fourth reduction showed 60 units and after regrinding 98 units. This appears to be due largely to reduction in the size of particles.

Investigations of Mill Streams.—Richardson (1884) published some of the earliest data available on the composition of mill streams and finished flours from several sections of the United States. His very extensive series of analyses were reported as to: moisture, ash, oil, carbohydrate, fiber, albuminoid nitrogen, phosphoric acid and gluten content. Baking tests were likewise reported; the yield of bread from a given weight of flour was given the most consideration in the analysis of the results.

Bailey (1925) presented the analyses made by Jacobs of all the mill streams of a certain mill.

Analyses including chemical composition and baking tests on the mill streams of a five-break mill were reported by Swanson, Willard, and Fitz (1915). The results obtained by these workers show clearly the excellent baking qualities possessed by the break flours.

Weaver (1921) examined a series of mill streams with respect to ash, gluten, loaf volume, and color. No relation between ash content and gluten could be distinguished in this study. Weaver also concluded that the condition of the wheat before milling and the subsequent mill manipulation were more responsible for variations in ash content than the original ash content of the wheat.

Hendel and Bailey (1924), using the viscosity test, measured the gluten quality of the various mill streams. Dividing the product of the crude protein content, and gluten quality factor by the product of the loaf volume times a texture factor gave, in most instances, a reasonably constant value. Exceptions were accounted

for either by a low-quality, high-gluten content or abnormal diastatic activity.

In a recent study by Cairns and Bailey (1928) the proteolysis of flour-in-water suspensions of different mill streams was determined. Amino nitrogen of an autolyzed suspension was determined using the Sørensen titration. The breaks and tailings flours showed considerably more proteolysis than the more refined streams. The same samples were also studied with respect to viscosity changes in suspensions after 5 hours digestion. A significant positive correlation between decrease in viscosity and increase in amino nitrogen was computed from these data.

Flour Proteins and the Lyotropic Series.—The lyotropic or Hofmeister (1888) series, showing relative neutral salt effects on colloidal systems, has been the subject of a voluminous literature. The general relations of the phenomena to colloid chemistry have been discussed at length by Bancroft (1918, 1921), Freundlich (1926), Rideal (1926), and Gortner (1929). Loeb (1921, 1922) argued against the existence of lyotropic effect of the various ions on protein systems.

Hoffman and Gortner (1927) were the first to note the differential solubility or peptization of flour proteins in different salt solutions. The method in common usage, using 5% K_2SO_4 or 10% NaCl solutions, tacitly assumed an equal solubility of the "globulin" fraction of wheat flour in these solutions. The final products isolated, using the two solutions mentioned as "solvents," differed greatly as to: amount obtained, chemical constitution as revealed by the Van Slyke method of analysis, and ease of redissolving in the original salt solution. A more extensive investigation was conducted later by Gortner, Hoffman and Sinclair (1928, 1929) in which 12 flours were extracted with 21 different inorganic salt solutions, using 0.5, 1.0, and 2.0 N solutions of most of the salts. The results show a distinct lyotropic series for both anions and cations which, with but few reversals, maintained the usual order within the series. The potassium halide solutions showed the most striking regularity with respect to the peptizing power of the halogen ions. Normal solutions of these salts extracted, on an average, KF 13%, KCl 23%, KBr 37%, and KI 64% of the total protein in the flour. Furthermore it was shown that the concentration of the salt solution had only a small effect, in most cases, on the peptizing action; that H-ion concentration played no part in accounting for the varying amount of protein peptized by the different ions

and, lastly, that no hydrolysis of the protein could be detected. It was concluded, from all of the evidence, that the existence of a wheat-flour globulin, as a distinct entity, was seriously open to question.

A statistical study of the above data made by Gortner (1927) revealed the fact that a significant negative correlation existed between the loaf volume and the percentage of protein extractable with a salt solution. The highest coefficient obtained was $r = -0.925 \pm 0.028$ for the series using normal $MgSO_4$, and the lowest, $r = -0.672 \pm 0.107$, for the normal KI series. Calculation of the correlation coefficient between loaf volume and the extracted protein using 5% K_2SO_4 solution for the flours, studied by Sharp and Gortner (1923), gave essentially the same figure: $r = -0.824 \pm 0.065$. It was suggested that after further verification, this apparently trustworthy relation might be used in the prediction of loaf volume without resorting to the time-consuming baking test.

Experimental

The Problem.—The problem as presented was conducted along two principal lines of investigation:—First, the study and comparison of the properties of wheat flour milled from the same sample of wheat with the commercial size of mill (The Minnesota State Experimental Flour Mill), and with the small experimental flour mill; second, the general study of the various mill streams of the milling system employed at the Minnesota State Experimental Flour Mill.

Published evidence, concerning the assumption that experimentally milled flour is identical with flour commercially milled, is meager and conflicting. Such a direct comparison is, however, a common laboratory and trade practice.

In the general study of the mill streams, special emphasis was given to an investigation of the differential peptization of the proteins of these flours by solutions of magnesium sulfate and the potassium halide salts. It was considered worth while to attempt to confirm the results obtained by Gortner, Hoffman, and Sinclair (1928, 1929), using flours showing more variation in protein content and physical properties.

1. **The Material.—Origin and Description of Wheats.**—The wheats from which the flours used in this investigation were milled were selected for milling tests at the Minnesota State Experimen-

tal Flour Mill. The procedure for the milling tests has been described by Bailey (1923).

No. 387 A hard red spring wheat from Carlos, Minnesota. Grade: No. 1 dark northern spring, 2% dockage, test weight 58.5 pounds per bushel.

No. 392 A hard red spring wheat from Clarkfield, Minnesota. Grade: No. 3 dark northern spring, no dockage, test weight 56.5 pounds per bushel.

No. 401 A hard red spring wheat from Shawmut, Montana. Grade: No. 2 northern spring, 1% dockage, test weight, 62 pounds per bushel.

No. 423 Marquis wheat from Manitoba Experimental Farm, Brandon, Manitoba. Grade: No. 4 northern spring, no dockage.

No. 424 Ceres wheat from Manitoba Experimental Farm, Brandon, Manitoba. Grade: No. 1 northern spring, no dockage.

No. 426 A blend of 55% of a hard red spring wheat from Lindsey, Montana. Grade: No. 1 dark northern spring, 2% dockage, test weight 59 pounds per bushel; 45% of a hard red spring wheat from Butte, North Dakota. Grade: No. 2 dark northern spring, 3% dockage, 2.6% durum; test weight 58.5 pounds per bushel.

2. **The Flour Samples.**—Wheat, and patent and straight-grade flour samples were collected from each of the milling tests at the Minnesota State Experimental Flour Mill. The wheat samples were then milled, using laboratory experimental flour mill producing a 75% patent flour.

The eighteen flours thus obtained, together with a series of samples from seventeen mill streams from the State Experimental Flour Mill, constituted the materials studied in this investigation.

The mill-stream flours were included in this study because they, too, represent flour resulting from different milling treatment and, incidentally, vary considerably in chemical composition and physical properties.

Methods

1. **Experimental Milling.**—Two thousand grams of cleaned wheat was weighed in a tightly covered container. Sufficient water was added to raise the moisture content to 14.5%, the sample thoroughly mixed, and allowed to stand. After 12 hours the wheat was retempered by adding water, until 15.5% of moisture was reached, and again the mixture was allowed to stand for three hours.

In the initial grinding the first and second, third and fourth, and fifth and sixth "breaks" were made, respectively, with the Nos. 16, 20 and 24 corrugated rolls. The bran was collected on a No. 20 wire mesh sieve, the shorts on a No. 30 GG cloth sieve, and the middlings were collected on a No. 12 XX cloth, the flour passing the No. 12 XX comprising part of the clear grade flour. The shorts and bran were passed through the smooth rolls once, and

sifted to reclaim adhering flour, which was added to the clear grade.

Eight reductions of the middlings were carried out, using the smooth rolls. The middlings were purified during the reduction process by passing them successively through No. 50 GG, No. 64 GG, and No. 70 GG cloth sieves, regrinding, each time, the middlings retained on the No. 10 XX sieve. Flour passing the No. 10 XX was combined, re-sifted and reserved as patent flour.

2. **Diastatic Activity.**—The diastatic activity of the laboratory mill patent and the large mill patent and straight-grade flours was determined, using Rumsey's method, as modified slightly by Sherwood and Bailey (1926).

Ten-gram samples were weighed into 200 cc. volumetric flasks and placed in a water bath which was held at a temperature of 27°±0.1°C. After attaining the temperature of the bath, 100 cc. of distilled water, warmed to 27°C., was pipetted into flasks and the flasks rotated to produce a smooth suspension. After digestion for exactly one hour, enzyme action was inhibited and the suspension clarified by the addition of 3 cc. of 15% sodium tungstate and a few drops of concentrated sulfuric acid. The proper acidity for good clarification was attained by adding a slight excess of acid beyond the amount necessary to produce a pink color in the flour suspension, using thymol blue as indicator. After diluting to 200 cc. and shaking, the suspension was centrifuged at high speed for 10 minutes. Fifty cubic centimeter aliquots were then pipetted into 400 cc. beakers and the reducing sugars determined by the Quisumbing and Thomas (1921) modification of the Fehling copper reduction method. With this method the Fehling's solution and sugar mixture is heated for exactly 30 minutes in a water bath at 80°C.

After the reduction period the Cu_2O was collected on asbestos in Gooch crucibles and the copper determined by the volumetric permanganate method. This procedure consists of transferring the asbestos mat and Cu_2O to a beaker and adding 50 cc. of a hot 20% H_2SO_4 solution previously saturated with $Fe_2(SO_4)_3$. The mixture is then titrated, while hot, with a standard solution of $KMnO_4$. The $KMnO_4$ solution used had a Cu equivalent of 10.00 mgm. per cc. The weight of reducing sugars, in terms of maltose, were obtained by consulting the tables published by Quisumbing and Thomas (1921). The blank for the reagents was combined with the blank for the natural reducing sugars in the flour, necessitating

only a single correction to obtain the weight of maltose produced by diastatic action.

In the study of the diastatic activity of the mill stream flours, the method of procedure was essentially the same as that noted above. It seemed desirable, however, to eliminate any variation that might occur due to differences in H-ion concentration of the flour-in-water suspensions. This was accomplished, in most instances, by the addition of 5 cc. of a sodium acetate-acetic acid buffer, having a pH of 5.0. In the case of the lower grade flours with a high natural buffer capacity, a predetermined amount of 0.05 N lactic acid was added to bring the pH of the digestion mixture to approximate pH 5.0.

3. **H-ion Concentration.**—The H-ion concentration of flour-in-water suspensions was determined in all cases by the electrometric method. Five grams of flour was transferred to a centrifuge tube and 50 cc. distilled water added. The tube was stoppered and shaken to produce a uniform suspension. The suspension was shaken at frequent intervals during one hour. At the end of this time the water extract was clarified by centrifuging for 10 minutes. The potentiometric readings were taken as soon as possible, using a Leeds and Northrup precision potentiometer graduated to read directly in pH. The Bailey hydrogen electrode was used, and each determination was duplicated, using two different electrodes.

In determining the pH of the buffered flour-in-water suspensions of the mill-stream flours, 2.5 cc. of the sodium acetate-acetic acid buffer was added and the suspension digested for an hour.

To determine the quantity of 0.05 N lactic acid necessary to produce an acidity of pH 5 in suspensions of some of the low-grade mill streams, a bubbling hydrogen electrode of the Hildebrand type was successfully employed. The 0.05 N lactic acid was added directly to the flour suspension contained in a titration vessel and the potentiometric reading was taken after each addition of acid. Each reading was duplicated, using two different electrodes.

4. **Baking Tests.**—The baking tests were conducted according to the standard 100 gm. flour procedure recently adopted by the Committee on Standardization of the Experimental Baking Test of the American Association of Cereal Chemists (Blish, 1928). The loaf volume was measured, using the measuring device designed by Werner. Each of the 36 flours was baked in duplicate on different days.

5. **Protein Extractions with Salt Solutions.**—The method followed in studying the differential peptization of flour proteins by different salt solutions was essentially the one described by Gortner, Hoffman, and Sinclair (1928). A preliminary study of the relative proportions of the peptizable protein removed by successive extractions on the same sample, showed that the number of extractions per sample might be reduced to one or two. Accordingly, the method adopted involved the use of two successive extractions for each sample.

Six-gram portions of flour were placed in a series of thick-walled, non-lipped centrifuge tubes of 100 cc. capacity. To these was added 50 cc. of the salt solution, and the stoppered tube shaken to suspend the flour in the liquid. The tubes then were placed in a mechanical shaker and shaken for a half hour after which the flour suspensions were centrifuged at high speed for 10 minutes. The clear or slightly opalescent liquids were decanted into 800 cc. Kjeldahl flasks, an additional 50 cc. of salt solution was added to the residue in the centrifuge tubes, the solid mass was loosened and broken up with a stirring rod, and the tubes were again shaken for one-half hour. The solutions from the second extraction were combined with those of the first extraction and the nitrogen determined by the Kjeldahl-Gunning procedure. Each of the determinations was conducted in triplicate, and if there was poor agreement between replicates, the procedure was repeated.

Moisture and total nitrogen determinations were made on the flours, and the protein content (N x 5.7) calculated on a moisture-free basis.

The salt solutions were prepared in all cases from "C. P." grade chemicals and the final solutions standardized. Since Gortner et al, 1928, 1929) found that the effect of concentration on the peptizing power of solutions of many of the salts was small, and the results for 0.5 N concentrations were, in general, representative, a single solution of each salt of 0.5 N concentration was chosen for this work.

The Data

1. **Mill Yields.**—The yields of the different mill products, obtained by grinding each of the wheats in the small experimental flour mill, are shown in Table I. The shorts and bran were combined as total feeds and recorded as the per cent by weight of the original weight of the wheat sample. The weight of the "clear"

TABLE I

SHOWING RESULTS OF MILLING TESTS ON DIFFERENT WHEATS

Wheat Number	Laboratory Mill				Commercial Mill[1, 2]		
	Total Flour	Total Feeds	Total	Yield of 75% patent in gms. from 2000 gms. wheat	Total Flour[3]	Total Feeds	Total Products
	%	%	%		%	%	%
387	66.00	34.00	100.00	960	68.53	33.84	102.37
392	69.50	30.50	100.00	913	69.47	34.10	103.57
401	69.25	30.75	100.00	1009	73.03	27.55	100.58
423	71.00	29.00	100.00	960	73.38	28.05	101.43
424	70.00	30.00	100.00	998	72.25	29.07	101.32
426	71.00	29.00	100.00	935	.....	.....	.....

[1] Experimental data made available from the records of the Minnesota State Experimental Flour Mill.
[2] All yields calculated to basis of original moisture content of wheat.
[3] Total flour yields all corrected to basis of 13.5% moisture content in flour.

flours, combined with that of the patent, was recorded as total flour.

For purposes of comparison, the mill yields from the same wheats, as obtained from the milling procedure of the Minnesota State Experimental Flour Mill, are likewise shown in Table I. The total flour yield values are recorded as corrected to a uniform 13.5% moisture basis. Since wheat No. 426 for the laboratory milling tests was obtained from a "blended" wheat mixture, no information regarding the yields of milled products from the commercial mill was available.

2. **Saccharogenic Activity of "Commercial" and "Experimental" Mill Flours.**—The results from the determination of saccharogenic activity of the large mill patent and straight-grade and experimental mill patent flours are recorded in Table II. The digestions in each case were carried out at the natural H-ion concentration of the 1:10 flour-in-water suspensions. The pH of the individual flour suspensions are shown in the table.

TABLE II

COMPARISON OF SACCHAROGENIC ACTIVITY OF THREE SAMPLES OF WHEAT FLOUR MILLED FROM THE SAME SAMPLE OF WHEAT, AND THE HYDROGEN ION CONCENTRATION OF A 1:10 FLOUR-IN-WATER SUSPENSION

No.	Commercial Mill Patent		Experimental Mill Patent		Commercial Mill Straight-grade	
	pH	Maltose	pH	Maltose	pH	Maltose
		mgm.		mgm.		mgm.
387	6.05	137.3	6.03	52.7	6.12	110.9
392	5.90	112.8	5.83	39.5	5.83	103.4
401	6.00	171.1	5.95	41.4	6.29	163.6
423	6.30	120.4	6.23	33.8	6.37	110.9
424	6.30	146.7	6.14	91.2	6.28	125.1
426	6.03	163.7	6.01	37.6	6.12	149.5

An inspection of Table II reveals the fact that the commercially milled patent flours had, in all cases, a much higher saccharogenic activity than either the straight-grade or the experimentally milled flours. The differences range from only a slight advantage over the straight-grade to values of from 1.5 to over four times the saccharogenic activity of the "experimental" patents. The table shows that only a small part of the variations can be accounted for on the basis of differences in H-ion concentration. It is true that Rumsey (1922) found the saccharogenic activity to change rapidly with changes in H-ion concentration within the range of pH encountered in this study, yet the variations in saccharogenic activity which were found are of sufficient magnitude to place them outside the realm of influence of this factor.

The other alternative is to explain the wide difference in saccharogenic activity on the basis of differences in granulation or fineness of division of the flour particles. Alsberg and Griffing (1925), and Alsberg (1927) indicate that this factor is of importance in its bearing on the ease with which diastase may attack the starch granules in flour. Alsberg et al concluded, from a study of the effects of overgrinding on flour, that the increase in saccharogenic activity with increased fineness of granulation was due to two factors. The first, and most important, was the increased ease of dispersion of the starch in cold water, and the second, a mechanical factor, the freeing of the starch granules from the gluten matrix.

A cursory physical examination of the flour milled with the experimental mill convinces one that the degree of subdivision is considerably less than that of the commercially milled flours.

Accordingly, patent flour No. 401 was milled in a porcelain ball mill for varying periods of time and the saccharogenic activity was determined. These data are given in Table III and show that a flour with relatively high initial saccharogenic activity can have its activity materially increased by further milling. This, in gen-

TABLE III

SHOWING INCREASE IN SACCHAROGENIC ACTIVITY OF A FLOUR DUE TO GRINDING IN A BALL MILL FOR DIFFERENT PERIODS OF TIME

Time of Grinding in Ball Mill	Anhydrous Maltose by Diastase per 10 grams Flour	Increase by Milling	Increase Due to Regrinding
Hours	mgm.	mgm.	%
0	171.2	0	
4	182.6	11.4	6.6
8	208.9	37.7	22.0
20	231.4	60.2	35.2

eral, agrees with the results obtained by Alsberg and Griffing (1925) wherein the saccharogenic activity of a flour was increased fourfold by grinding 53 hours in a ball mill.

The variations exhibited between flours of the same class are typical when compared with the data of other workers. Hermano and Rask (1926) have recently published the results of a study of the susceptibility of different wheat starches to hydrolysis by diastase and suggested that individual variations might be traced to differences in geographical origin and climatic environment of the parent wheats.

3. **Saccharogenic Activity of the Mill Streams.**—The results for the saccharogenic activity of the mill-stream flours, together with the pH of the digestion mixtures are given in Table IV.

TABLE IV

SHOWING SACCHAROGENIC ACTIVITY OF THE VARIOUS MILL-STREAM FLOURS AT THE SAME H-ION CONCENTRATION

Arbitrary Grade	Mill Stream	Buffered Water Suspension	Anhydrous Maltose from 10 gms. Flour
		pH	mgm.
—	First break	5.11	299.1
+	Second break	5.10	230.5
+	Third break	5.18	186.3
—	Fourth break	5.13	203.2
—	Fifth break	5.17	197.6
++++	First middlings	5.08	264.3
++++	Second middlings	5.06	255.7
++++	Third middlings	5.06	291.6
+++	Fourth middlings	5.06	239.2
++	Fifth middlings	5.06	199.5
+	Sixth middlings	5.06	260.6
+	Sixth middlings tailings	5.06	211.6
—	First tailings	5.02	277.5
—	Second tailings	5.00	294.4
++	Sizings	4.95	354.6
- - -	Bran and shorts duster	5.05	336.7
- - -	Low grade	4.90	306.6

The values recorded are the averages of two or more replicates. The various flours used in this study were assigned arbitrary grades based upon their ash content. The plus signs were used to indicate relative degrees of refinement, and the minus signs the lower grades. In general, the high values for the saccharogenic activity are found in the lower grades of flour. Bailey (1923) reported the per cent of ether extract in the various mill streams of the Minnesota State Experimental Flour Mill, as an index of the germ content of the flour. In the light of these data, the high saccharogenic activity of the low-grade, duster flour, sizings, first

and second tailings, and fourth and fifth "break" flours may be attributed to a relatively high germ content.

4. **Baking Tests on "Commercial" and "Experimental" Flours.** —The complete data obtained from the baking tests on the "commercial" and "experimental" flours are given in Table V. The different values there recorded were obtained from the average of duplicate determinations baked on different days. The mean values for the different types of flour are shown for purposes of general comparison. An inspection of the percentage "absorption" reveals

TABLE V

RECORD OF PROTEIN CONTENT OF FLOURS AND COMPARATIVE BAKING TESTS WITH COMMERCIALLY AND EXPERIMENTALLY MILLED FLOURS

No.	Description	Crude Protein (Moisture-free basis)	Absorption	Volume of loaf	Color Score	Texture	Grain	Crust Color
		%	%	cc.				
387	Pat.	12.37	63	372	98	98.0	96.0	98.5
387	Str.	12.54	63	397	96	99.0	94.5	98.0
387	Exp.	11.86	53	407	98.5	98.0	98.5	98.5
392	Pat.	13.34	64	430	98	97.5	98.5	97.0
392	Str.	14.48	63	450	98	97.5	97.0	97.0
392	Exp.	14.08	53	442	99	97.0	98.0	98.5
401	Pat.	13.22	65	430	100	97.0	99.0	97.5
401	Str.	14.08	68	417	99.5	99.0	99.5	99.0
401	Exp.	12.97	54	402	100.5	97.5	99.0	98.5
423	Pat.	11.91	64	377	98	98.0	98.0	96.5
423	Str.	12.43	65	362	98	98.5	97.0	98.5
423	Exp.	11.86	55	347	98	98.0	98.0	97.5
424	Pat.	12.54	71	362	99	98.0	96.5	98.0
424	Str.	13.17	73	392	98	98.0	95.0	96.5
424	Exp.	12.31	58	360	98	97.0	98.0	99.0
426	Pat.	13.17	63	400	99	97.5	99.0	98.5
426	Str.	13.39	63	400	98	98.0	98.0	98.5
426	Exp.	12.65	56	382	99	98.0	98.5	98.5
	Mean for patent	12.76	65	395.2	98.5	97.6	97.8	97.6
	Mean for Straight grade	13.35	65.8	403.0	97.9	98.8	96.8	97.8
	Mean for Experimental	12.62	54.8	390.0	98.8	97.6	98.3	98.4

Pat. = Patent; Str. = Straight; Exp. = Flour milled on laboratory mill.

the fact that the absorption of the "experimental" patent flours is invariably lower than the corresponding values for the "commercial" flours. The mean values show a difference of approximately ten per cent with respect to this property. No consistent variations were encountered in the loaf volume representing the three flour types. The differences in the mean values probably have no significance in view of the number of observations involved. In view of the personal element involved in the arbitrary assignment of values to the other bread properties, little significance can be

attached to the small differences obtained in the color, texture, grain, and crust color scores. Considering the average values, the "commercial" and "experimental" patents are virtually identical as to color score and texture and differ only slightly in grain and crust color scores. As would be expected, the straight-grade flour is slightly inferior as to color and grain, but superior as to loaf volume and texture, and intermediate in crust color value.

5. **Baking Tests on Mill Streams.**—Table VI shows the results of the experimental tests of the seventeen mill-stream flours. The

TABLE VI

RECORD OF PROTEIN CONTENT OF FLOURS AND COMPARATIVE BAKING TESTS ON SEVENTEEN MILL STREAM FLOURS

Description	Crude Protein (Moisture-free basis)	Absorption	Volume of loaf	Color Score	Texture Score	Grain Score	Crust Color Score
	%	%	cc.				
First break	13.62	61	387	89.0	98.5	97.5	98.5
Second break	13.74	60	372	95.5	99.0	98.5	98.0
Third break	15.79	62	430	93.5	98.0	99.0	97.5
Fourth break	17.50	62	475	90.0	97.0	98.0	97.0
Fifth break	19.55	63	487	87.0	96.0	97.0	97.0
First middlings	13.11	62	347	97.0	98.0	97.5	98.0
Second middlings	12.71	62	390	98.0	98.0	98.0	98.0
Third middlings	12.82	63	385	98.0	98.5	98.5	98.0
Fourth middlings	13.28	63	400	99.5	98.0	99.5	97.0
Fifth middlings	13.28	62	415	99.5	98.0	99.5	97.5
Sixth middlings	13.79	65	432	99.0	97.0	98.5	97.5
Sixth middlings tailings	13.34	64	417	99.0	98.0	99.0	98.0
First tailings	12.54	66	372	96.0	91.0	93.5	98.0
Second tailings	14.02	64	412	94.0	86.0	94.0	98.0
Sizings	12.48	62	380	96.5	98.0	97.0	100.0
Bran and shorts duster	15.39	60	415	82.5	80.0	92.5	97.0
Low grade	22.34	73	310	70.0	70.0	50.0	95.5

values for the loaf volumes are highly variable. It is notable also that many of the flours graded low in Table IV show, in general, the larger loaf volumes. An examination of the color scores, however, serves to point out the inferiority of these flours. The texture, grain, and crust color scores are somewhat less variable in magnitude but are in most cases indicative of flour quality. The absorption values, it should be noted, fall within a relatively narrow range.

6. **Flour Proteins and the Lyotropic Series.**—Gortner, Hoffman, and Sinclair carried out three extractions with fresh portions of the salt solution on each sample. Since this was a rather laborious and time-consuming procedure, a preliminary study was un-

dertaken to ascertain whether a single extraction could be substituted for the triple extraction. Table VII shows the results obtained from the nitrogen determinations on the first extract, on the second plus the third extracts, and on the fourth plus the fifth extracts from the same sample of flour. The results are given for extractions with 0.5N KBr, KCl, and $MgSO_4$ solutions, using three flours which differed somewhat in total nitrogen content.

The first column gives the per cent of total nitrogen for the different flours, and Columns 3, 6, and 9 show the nitrogen in milligrams which was peptized by the successive treatments with salt solutions. Columns 4, 7, and 10 give the percentages of the total nitrogen of the flour peptized in the several fractions. In Columns 5, 8, and 11 are shown the nitrogen found in the extracts expressed in terms of per cent of the total nitrogen which was peptized. Thus, the nitrogen in each fraction is shown in its relation to the total nitrogen of the flour and to the total peptizable nitrogen. If a single extraction were to give the same relative peptization values for different salt solutions as the method employing three extractions, the peptizable nitrogen in the first fraction, expressed either as percentage of total nitrogen or of total peptizable nitrogen, should, using a given salt solution, assume a constant value within the series.

After a consideration of the experimental errors involved, and of the inherent differences between flours encountered by Gortner et al and later in this work, the values obtained show fair agreement.

Because of the variations shown in the preliminary study, it was thought that in subsequent work some of the irregularities might be eliminated by making two extractions on the same sample, at the same time effecting a substantial saving in time over the method as used by Gortner and his co-workers. The percentage error in the nitrogen determination would also be reduced by the addition of the nitrogen peptized by a second treatment of the sample.

The results of the study of the relative amounts of nitrogen peptized from the flours of the seventeen mill streams by two extractions with 0.5 N solutions of KBr, KCl, KF, and $MgSO_4$ have been assembled in Table VIII. It was assumed here that all of the nitrogen was of protein origin, but in the consideration of the data nothing was to be gained by converting it into terms of protein by using the arbitrary conversion factor, 5.7. The nitrogen peptized by the different salt solutions was expressed in each case as the

TABLE VII

SHOWING RELATIVE AMOUNTS OF NITROGEN PEPTIZED BY SUCCESSIVE TREATMENTS WITH 0.5N SOLUTIONS OF KBr, KCl, AND $MgSO_4$

Total Nitrogen in Flour	Description	0.5N KBr			0.5N KCl			0.5N $MgSO_4$		
		Nitrogen Peptized	Per Cent of Total Nitrogen	Per Cent of Total Nitrogen Peptized	Nitrogen Peptized	Per Cent of Total Nitrogen	Per Cent of Total Nitrogen Peptized	Nitrogen Peptized	Per Cent of Total Nitrogen	Per Cent of Total Nitrogen Peptized
%	Flour A	mgm.			mgm.			mgm.		
1.62	Extract 1	21.78	25.71	65.17	16.28	19.23	66.12	15.18	17.86	76.09
	Extracts 2 and 3	9.46	11.17	28.31	5.61	6.62	22.79	3.87	4.57	19.40
	Extracts 4 and 5	2.18	2.57	6.52	2.73	3.22	11.09	0.90	1.06	4.51
	Total	33.42	39.46	100.00	24.62	29.07	100.00	19.95	23.56	100.00
2.43	Flour B									
	Extract 1	26.85	21.10	63.58	19.50	15.32	71.30	19.07	15.18	76.38
	Extracts 2 and 3	12.24	9.61	28.98	5.00	3.93	18.28	4.04	3.17	16.16
	Extracts 4 and 5	3.14	2.47	7.43	2.85	2.24	10.42	1.89	1.48	7.56
	Total	42.23	33.17	100.00	27.35	21.48	100.00	25.00	19.63	100.00
2.19	Flour C									
	Extract 1	25.50	22.00	65.02	19.30	16.39	71.35	18.89	16.29	76.97
	Extracts 2 and 3	9.75	8.41	24.86	5.47	4.72	20.19	4.24	3.66	17.28
	Extracts 4 and 5	3.97	3.42	10.12	2.32	2.00	8.56	1.41	1.22	5.75
	Total	39.22	33.83	100.00	27.09	23.37	100.00	24.54	21.17	100.00

TABLE VIII

SHOWING RELATIVE AMOUNTS OF NITROGEN PEPTIZED FROM THE MILL-STREAM FLOURS BY TWO EXTRACTIONS WITH 0.5 N SOLUTIONS OF KBr, KCl, KF, AND $MgSO_4$

No.	Sample	Nitrogen in Flour Moisture-free Basis	Total Nitrogen in Flour Peptized by:			
			0.5 N KBr	0.5 N KCl	0.5 N KF	0.5 N $MgSO_4$
		%	%	%	%	%
1	First break	2.39	27.16	18.97	14.69	18.51
2	Second break	2.41	23.63	17.19	13.41	16.89
3	Third break	2.77	23.45	16.65	12.57	15.83
4	Fourth break	3.07	25.86	19.00	14.00	18.37
5	Fifth break	3.43	28.11	21.69	15.67	20.28
6	First middlings	2.30	26.88	18.70	13.79	17.56
7	Second middlings	2.23	27.18	19.21	14.28	17.95
8	Third middlings	2.25	26.40	18.89	14.06	17.46
9	Fourth middlings	2.33	26.53	18.21	13.64	17.01
10	Fifth middlings	2.33	26.64	19.32	14.06	17.89
11	Sixth middlings	2.42	27.14	19.41	14.54	17.95
12	Sizings	2.19	28.39	20.71	15.52	19.90
13	Sixth middlings tailings	2.34	29.68	21.87	16.28	20.27
14	First tailings	2.20	28.67	23.98	17.67	22.97
15	Second tailings	2.46	36.81	29.21	22.23	28.35
16	Low grade	3.92	34.17	29.93	26.86	28.20
17	Bran and shorts duster	2.70	34.57	27.99	24.08	25.82
	Average		28.31	21.23	16.31	20.07

per cent of the total nitrogen content of the respective flours. The average values for the entire series of flours were calculated and appear in the table.

Discussion of the Data

1. **Saccharogenic Activity.**—From the results of this comparative study of the saccharogenic activity of commercially and experimentally milled flours, it is apparent that flours milled with the laboratory mill do not provide reliable information regarding the saccharogenic properties of commercially milled flours milled from the same wheats. While the present series of data are obviously meager for generalization, this contention is supported by additional data (yet unpublished) collected by one of the authors (Sherwood). The discrepancies which were observed cannot be accounted for on the basis of differences in H-ion concentration. Since the temperature and time of reaction were kept constant, the differences in size of particles remain as the probable principal causal factor. This difference in degree of subdivision is a variable which seemingly has received little consideration by workers in the field and although probably relatively unimportant in the comparative study of "commercial" flours, it should be considered as one of the reaction variables.

Contrary to the results of Rumsey and others the apparent deficiency in saccharogenic activity of the "experimental" flours was not reflected in the loaf volume when the flours were baked. The average loaf volumes for the different flours, shown in Table V, are essentially identical, yet the saccharogenic activity of the "commercial" flours ranged from 1.5 to 4 times that of the "experimental" flours. This lack of correlation between loaf volume and saccharogenic activity may be attributed to two factors: the quantity of sugar added to the dough and the relatively short, *standardized* fermentation period employed in the "small dough" baking test. The quantity of sucrose added in the dough formula evidently was sufficient for the nutrition of the yeast during the fermentation period and did not need to be augmented greatly by sugars formed by starch hydrolysis.

Judging from these results, the basic procedure of the American Association of Cereal Chemists standard baking test, tentatively adopted, fails to distinguish in terms of loaf volume such marked differences in saccharogenic activity, as were represented by the flours used in this study. It is conceivable that a commercial procedure involving a longer fermentation period and less sugar in the formula might give quite different results from those given by the standard baking test.

The American Association of Cereal Chemists has added a modification of the basic procedure providing for longer fermentation periods. This supplementary method has been added since the present study was completed, and it has been suggested that it may be used when it is desirable to determine fermentation tolerance.

Blish, Sandstedt, and Platenius (1929) have shown a significant positive correlation between diastatic activity and crust color, also between residual sugar in the loaf and crust color based on tests with the standard A.A.C.C. baking method. From their results and the data presented here it is quite evident that crust color and not loaf volume should be the criterion of diastatic activity when the A.A.C.C. baking test with fixed fermentation time is used.

If any significance were to be attached to the "absorption" of the flour, the values shown in Table V clearly indicate that the experimentally milled flours do not give a reliable index to absorption of commercially-milled flours.

The saccharogenic activities of the various mill-stream flours determined at a constant H-ion concentration show a considerable range of variation. The results given in Table IV show the effect

of composition and to some extent the effect of mechanical treatment on the saccharogenic activity of the flours. As might be expected the saccharogenic activity is particularly indicative of the germ content of the various streams. This fact has been pointed out many times by other workers.

2. **Baking Tests.**—The only significant difference between the "commercial" and "experimental" flours revealed by the baking test was the consistently lower "absorption" of the "experimental" flours. The experimental flours had a mean absorption approximately 10 per cent below the mean absorption value for the commercial flours.

In view of the consideration at present being given to the relation between total protein content and loaf volume, it seemed desirable to test the relation between these variables in the "commercial" and "experimental" series of flours. Such a relation is best expressed by the coefficient of correlation. Accordingly, the data for protein content and loaf volume as presented in Table V were subjected to a statistical study, using the formula developed by Harris (1910). The coefficient of correlation between loaf volume and protein content was found to be $r = +0.797 \pm 0.041$. This constant which is roughly twenty times its probable error may be considered unquestionably significant. The class averages representing protein content and loaf volume were plotted graphically. The regression line was likewise plotted and showed that in this series of flours the relation between protein content and loaf volume was approximately linear.

From the data for the mill-stream flours in Table VI the correlation coefficients between the same variables were calculated. The coefficient of correlation between protein content and loaf volume was found to be $r = -0.018 \pm 0.164$. It is of interest that this coefficient, when considered with respect to its probable error, is of no significance in expressing a relationship between the variables, if the series of flours comprises different mill streams milled from the same sample of wheat, as contrasted with a high correlation when the same grade of flour is milled from each of several different lots of wheat.

3. **Flour Proteins and the Lyotropic Series.**—The data presented showing the specific ion effect on the peptization of flour proteins confirm in all respects the findings of Gortner, Hoffman, and Sinclair (1928, 1929). From an inspection of Table VII and particularly of Table VIII, it is clear that a definite lyotropic effect

exists within the halogen salt series. Without a single reversal the ion effect may be arranged Br>Cl>F. The relative amounts of nitrogen peptized are less, with the exception of the values for KF, than those reported by Gortner et al. This is probably due in part to the reduction in the number of extractions made on each sample from three to two. It was thought that perhaps the decidedly abnormal values obtained for the second tailings, low-grade, and duster flours were exerting a disproportionate influence on the calculated means for the whole series. Accordingly, the means, standard deviations, and coefficients of variability were calculated for each salt, first with the whole series intact (N = 17), and then with the mill-stream flours Nos. 15, 16, and 17 excluded from the series (N = 14). The results appear in Table IX.

TABLE IX

THE MEAN, STANDARD DEVIATION FROM THE MEAN, AND VARIABILITY OF THE PERCENTAGES OF TOTAL NITROGEN PEPTIZED BY FOUR SALT SOLUTIONS FROM A SERIES OF MILL-STREAM FLOURS

No. in Series	Constant	KBr	KCl	KF	$MgSO_4$
17	Mean	28.31±0.58	21.23±0.65	16.31±0.65	20.07±0.62
17	Standard deviation	3.556037	4.006896	3.991330	3.803029
17	Coefficient of variability	12.56%	18.87%	24.46%	18.95%
14	Mean	26.84±0.30	19.56±0.34	14.58±0.23	18.49±0.32
14	Standard deviation	1.665326	1.864945	1.265120	1.755418
14	Coefficient of variability	6.20%	9.54%	8.68%	9.49%

From Table IX it is quite clear that the three abnormal members of the flour series have doubled and, in the case of the KF series, trebled the variability. The standard deviations from the means were likewise reduced approximately one-half by excluding flours Nos. 15, 16, and 17. The table also reveals the fact that the percentages of total nitrogen peptized by 0.5 N KBr solution are less variable than the corresponding values for the other salt solutions.

In Tables VII and VIII the flours with high nitrogen content apparently yield a *relatively* larger peptizable fraction. An attempt was made, therefore, to measure the relation between total nitrogen content and the peptizable fraction. The most desirable means of expressing this relationship seemed to be by calculation of the coefficient of correlation between the nitrogen content and the deviation of the peptized fraction from its probable value. The following formula developed by Harris (1909) was used:

$$r_{pz} = \frac{r_{p\pi} - V_p/V_\pi}{\sqrt{1 - r_{p\pi}^2 + (r_{p\pi} - V_p/V_\pi)^2}}$$

r_{pz} = the correlation between the nitrogen content and the deviation of the peptized fraction from its probable value where:

$r_{p\pi}$ = the correlation coefficient between total nitrogen content and the amount of nitrogen peptized.

V_p = the coefficient of variability for the total nitrogen content.

V_π = the coefficient of variability for the amount of peptized nitrogen.

The calculated results are found in Table X.

TABLE X

SHOWING FOR A SERIES OF MILL-STREAM FLOURS THE CORRELATION COEFFICIENT BETWEEN THE TOTAL NITROGEN CONTENT AND THE AMOUNT OF NITROGEN PEPTIZED, AND THE CORRELATION BETWEEN THE NITROGEN CONTENT AND THE DEVIATION OF THE PEPTIZED FRACTION FROM ITS PROBABLE VALUE

0.5 N Salt Solution	$r_{p\pi}$	r_{pz}
KBr	+0.87707	+0.369316
KCl	+0.860561	+0.554965
KF	+0.83854	+0.606362
$MgSO_4$	+0.858840	+0.547994

The calculated coefficients, r_{pz}, show that the influence of the original protein concentration on the relative amounts peptized by the various salt solutions varies in the reverse order of the peptizing effect of the ions. In other words, with the KBr solution the peptized fraction more nearly approaches a constant value irrespective of the protein content of the flour than with any of the other salts used. Whatever the explanation may be for the variations here encountered, the data demonstrate again, in a slightly different manner, the lyotropic effect of the ions in question.

To determine whether the relation between loaf volume and the peptizable protein fraction, recently reported by Gortner (1927), could be demonstrated with the mill-stream flours, the coefficients of correlation between these variables were computed. For the KF series the correlation was $r = -0.317 \pm 0.147$; for the KCl series, $r = -0.235 \pm 0.072$; for the KBr series, $r = -0.186 \pm 0.158$; and for the $MgSO_4$ series, $r = -0.241 \pm 0.154$.

In the light of the number of observations involved and the magnitude of the probable errors, little significance can be attached to these correlation coefficients. The flours used were quite different in their physical and chemical natures from those used in the study reported by Gortner (1927). The gluten and non-gluten protein ratios in many of the mill streams were undoubtedly quite different from normal wheat flour. This is substantiated by the

observation that the correlation between loaf volume and protein content in the mill-stream flours is strikingly insignificant, whereas in normal flours there is commonly a high positive correlation. It seems probable that when there is no correlation between loaf volume and protein content, there will be little or no correlation between loaf volume and the percentage of protein peptized by any particular salt solution. It should be further noted that in these mill-stream flours not all of the nitrogen can be referable to protein, since undoubtedly portions of germ are present in certain of the mill streams which would introduce non-protein nitrogenous compounds, such as nucleic acid, etc. Strict comparison therefore between data involving normal patent or straight-grade flours and data involving all of the mill streams is not justified.

Summary and Conclusion

A comparative study of the properties of a series of "commercial" and "experimental" flours milled from the same wheats has been made. There was also included in this study a comparative study of the properties of the flours from the seventeen mill streams of the Minnesota State Experimental Flour Mill.

The data presented seem to warrant the following conclusions:

1. The saccharogenic activity of flour milled with the experimental mill does not truly represent the potential activity of commercially milled flour from the same wheat. The flours milled in a commercial mill have from one to four times the saccharogenic activity of flours milled from the same sample of wheat in a small "Experimental Mill."
2. The degree of granulation of flours is an important factor in the consideration of the saccharogenic properties, as is shown by the fact that grinding a sample of flour milled in the commercial mill in a ball mill for twenty hours increased the saccharogenic activity approximately 35 per cent.
3. The basic procedure of the A.A.C.C. experimental baking test, without extended fermentation periods, does not permit detection of flours low in saccharogenic activity (when saccharogenic activity is measured on a flour-water suspension by the Rumsey method) from loaf volume values, as evidenced by the fact that the "commercial" flours and the "experimental" flours, while differing materially in saccharogenic activity did not differ appreciably in loaf volume.

4. In the peptizing action of potassium halide salts on flour proteins, a definite lyotropic effect can be demonstrated. This series is KF>KCl>KBr. The coefficients of variability were calculated for these halide salts, and the KBr series, although showing the highest peptization, showed the lowest variability.
5. The series of "experimental" and "commercial" (patent and straight-grade) flours gave a coefficient of correlation of $r = +0.797 \pm 0.041$ between protein content and loaf volume. A similar correlation on the mill-stream series of flours, all milled from one sample of wheat, was $r = -0.018 \pm 0.163$.
6. The serious objection recently raised against the present definition of a "globulin" is entirely justified. The coefficients of correlation between loaf volume and the fraction peptized from the mill-stream flours by the salts were -0.186 ± 0.158 for the KBr series, -0.235 ± 0.072 for the KCl series, -0.317 ± 0.147 for the KF series and -0.241 ± 0.154 for the $MgSO_4$ series.
7. The correlation coefficients between total protein content and the per cent of protein peptized from the mill stream flours by the various salts varied from +0.839 to +0.877 and the correlation between a variable and the deviation of a dependent variable from its probable value were found to be +0.369 for KBr, +0.555 for KCl, +0.606 for KF and +0.548 for $MgSO_4$ solutions (0.5 N).

These last data indicate that while the fraction of the protein petized by a salt varies with the percentage of protein which is present, nevertheless there is less influence of the original protein content in the KBr series than in the case of the other salts.

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CONTRIBUTION TO THE KNOWLEDGE OF COLLOID-CHEMISTRY OF THE GLUTEN. II.

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(Received for publication May 25, 1929)

In a preliminary communication (1929) we emphasized the phenomenon that manifests itself by discharging a protein solution obtained by shaking gluten with acid. As soon as the discharging has reached a certain point we find a formation of little drops in the liquid, which can be plainly observed microscopically. We compared this phenomenon with the "separation"[1] of homogeneous liquid into two phases, and pointed out that sundry properties, appertaining to the last mentioned system, could be found in the case in question. Also that by adding to a solution thus "separated" a substance miscible with the two components, homogeneous mixing resulted. In this paper we will deal with this phenomenon and will try to get a closer insight into it viscosimetrically. When studying such a process in a protein solution, in which are several proteins, there may be large complications. Therefore the system was kept as simple as possible and the study was strictly limited to only one of the proteins—gliadin.

At the same time we will show that there is a great probability that these phenomena associated with the "separation" do not manifest themselves suddenly at a certain rate of charge of the particles, but are rather a gradual transition of particles completely hydrated to particles with less hydration; in other terms, that in solutions microscopically void under maximal magnification, "separation" is already present. In close connection herewith we studied the behavior of gliadin solutions in different alcohol concentrations and tested the influence of changing H-ion concentration on an alcoholic gliadin solution.

We will point out further that the action of alcohol is diversified according to the concentration in the alcohol-gliadin sol.

Before proceeding to the method of purification of the gliadin used and the results of measurements with this product, it is desirable to refer briefly to the work done by Lüers on the colloid-chemical properties of gliadin; and the research work, recently

[1] Where the term "separation" is used in this paper it refers to the phenomenon of the formation of a new phase dispersed in an initial phase. Thus for example the initial system may consist of gliadin dispersed in water which may "separate" to a system of water-dispersed-in-gliadin which in turn is dispersed in water.

done by Kruyt and his collaborators of the University of Utrecht, regarding the behavior of emulsoids in alcohol-water solutions, and the stability factors of these groups of colloidal solutions. Lüers (1919) in his extensive researches on gliadin, experimented with solutions obtained by diluting definite quantities of a 70% alcoholic standard solution with water or electrolyte solutions. He studied extensively the influence of acid, alkali, and electrolyte solutions. We may, however, question some minor details of his researches. We doubt if the law of Poiseuille can be applied to some of the systems used by him. We feel that he must have meant to avoid this difficulty by using viscosimeters with a capillary diameter five times the normal. Also we object to the way he makes his protein solutions.

Lüers dilutes definite quantities of 70% alcoholic protein solution with varying quantities of aqueous electrolyte solutions. In his gliadin solutions a varying percentage of alcohol will appear, also the protein percentage in his solutions will vary. It must be difficult to apply a correction for these two variables, as the change effected in the viscosity by increasing the percentage of alcohol, or by increasing the percentage of protein, is not necessarily linear when depicted graphically. While assuming that these corrections can be properly applied, we say that for one definite series of determinations this may not be done. From what follows it appears that varying concentrations of alcohol of the same H-ion concentration have a decided influence on the relative viscosity of the gliadin solutions, and that by changing pH no comparison is possible. We suggest that the opinion of Lüers, that the alcohol sol shows an emulsoid character as against the suspensoid character shown by the hydrosol, is subject to objection. We can only explain the high viscosity of acid-water-gliadin solutions by assuming that the ϕ in the Einstein formula $\eta_s = \eta_0 (1 + K\phi)$ (electr. fact.) has a much larger value, as appears from the calculation with definite protein percentage for a suspensoid solution. We may assume that in an aqueous medium, gliadin shows an emulsoid character.

We will now briefly review what has lately been found viscosimetrically about the behavior of emulsoids in alcohol medium.

In his researches on the stability of agar solutions Bungenberg de Jong (1921) also studied the influence of alcohol on these solutions, arriving at the same conclusion as afterwards reached by Tendeloo (1926) using a gum arabic solution, namely that in the

above-mentioned solutions, definite percentages of alcohol exercise a dehydrating action on the emulsoid particle. It became possible thereby to remove one of the factors of stability of the emulsoid, namely, the water layer, and keep the particle stable only on its charge. While agar and gum arabic did not show a change of hydration upon changing the H-ion concentration, Lier (1924) experimented on casein, which undergoes a decided change of hydration upon changing the H-ion concentration. He investigated the influence of alcohol on the hydration of casein particles with maximum charge, on both the positive and the negative side of the isoelectric point. On both sides of the isoelectric point the phenomena differed. Casein charged negatively behaved like agar; when charged positively, no dehydration was found. Later we will refer to the mentioned researches, which have been dealt with only briefly.

We have still to mention the researches of Dill and Alsberg (1925). These authors showed that the dissolving of the prolamines in alcohol-water mixtures is not molecular but is more a peptization, as the solubility of these substances above certain critical temperatures (the critical peptization temperature, or C.P.T.) is unlimited.

Preparation of the Gliadin Fraction

It appears from the literature that the method of preparation of a protein fraction greatly influences the colloidal behavior of the product. It is therefore desirable to look into the method by which the preparation used by us has been obtained.

As Blish and Sandstedt (1926) advised, we took as a starting material well-washed gluten dried in vacuo; with this difference, that the drying temperature did not exceed 40°C. The higher temperature used by them we did not use because Sharp and Gortner (1923) found that drying gluten in vacuo at 45°-50°C. altered the colloidal properties of the gluten; Tague (1925) mentions that higher temperature alters the physical properties of gliadin. On the other hand it appeared to us that most vegetable proteins already lose their power to peptize easily by drying intensively at a low temperature, while gliadin showed little of this property. In this way the glutenin was made insoluble (Osborne points out that glutenin is slightly soluble in dilute alcohol) and at the same time a possible destruction of the gliadin by too high a temperature was avoided.

This finely powdered gluten was shaken for a long time with a large quantity of 70% alcohol, and was allowed to settle over night. After decantation the liquid was filtered clear. With the sediment, the method of shaking and settling was repeated.

The first and second alcoholic filtrates were combined. A third extraction of the sediment gave a milky, cloudy solution, which could not be clarified by filtration. This last solution was kept separate from the others. The clear filtrates were evaporated under reduced pressure—taking care that the temperature of 40° C. was not surpassed—until a syrupy substance began to settle out. After cooling the residue of evaporation, the dilute alcohol was separated from the tough syrup, which was put away to settle for a second time, and again the aqueous phase was poured off. Now the syrup was repeatedly washed with acetone, to get rid of fatty substances; at the same time tough strings were formed that were well washed with distilled water and subsequently with acetone. These protein strings were put away over night to harden in a large quantity of pure acetone. After thoroughly drying and powdering the protein, the entire operation was repeated twice, being particularly careful not to heat the alcohol used in the process. In order to remove the last traces of acetone, the preparation was finely powdered and dried at 30°C. The gliadin thus obtained was a white powder with a low ash content (0.09% ash on the dry material).

This gliadin was used in all subsequent experiments. It showed the same properties as the protein obtained by shaking gluten with acid.

The preparation was easily soluble in very dilute alkali or acid. By discharging an alkaline or acid gliadin solution, either by adding electrolytes or by changing the H-ion concentration, drops appear in the liquid that will pass through filter paper.

Upon carefully adding very dilute alkali to the acid protein solutions, a sticky substance settled out at the isoelectric point, which gathered into the foam upon shaking the liquid. Microscopically seen, the foam consists of many protein strings of a great number of globules partly deformed and strung together. We did not succeed in obtaining a precipitate that microscopically showed the fine structure of real flocculation by discharging the protein in aqueous solutions. Always the semi-liquid nature of the sediment remained.

In order to be sure no foreign substance that might be contained in the protein fraction promoted these phenomena of "separation," a part of the protein preparation was purified in different ways.

It was again dissolved in dilute alcohol and then precipitated out by adding absolute alcohol, which treatment was repeated twice, followed by dissolving in dilute acetone and precipitating again with 100% acetone.

These methods of purification do not alter the phenomenon of "separation"; neither does the washing out with 5% K_2SO_4 solution, nor afterwards washing thoroughly with distilled water, nor dissolving in dilute alkali and again precipitating with dilute acid.

Electrodialyzing of an acid gliadin sol made from a preparation thus purified does not alter the property of drop-forming upon discharging. This electrodialysis caused the following remarkable phenomenon. After one day of electrodialyzing, the protein solution reacted strongly alkaline as indicated by phenolphthalein, but on further dialyzing the reaction became entirely neutral and a part of the protein settled.

Finally we investigated whether fat or lecithin or such substances could be the cause of this "separation," but here also the result was negative. Extracting the protein in a Soxhlet apparatus with acetone and ether for two days gave no results.

We have not succeeded in avoiding the phenomenon of "separation" by extracting foreign substances from the protein preparation. In the face of this we may conclude that the property of "separation" is inherent to the protein fraction called gliadin.

MEASUREMENTS

Influence of Varying Concentrations of Alcohol on Positive Gliadin

The gliadin thus obtained was dissolved in the following way. This method of making solutions was applied to all the gliadin solutions used subsequently, unless otherwise stated. The total quantity of liquid was the same in all experiments (110 cc.), but varying quantities of acid were used.

Three grams of air-dried, finely powdered gliadin were shaken for one hour in a thermostat at 25°C. with 104 cc. distilled water and 6 cc. 0.1N HCl, a total of 110 cc. of liquid. After shaking one hour, all the substance was dissolved and the liquid was filtered.

This was used for the first series of experiments as a standard solution, and the influence of varying concentrations of alcohol on the viscosity of the protein solutions was investigated.

Varying quantities of alcohol were pipetted into a volumetric flask of 50 cc. and then distilled water was added, leaving room for 5 cc. of the standard gliadin solution. After adding 5 cc. of the protein solution, the volumetric flask was shaken and put in a thermostat at 25°C. After 15 minutes the liquid was made to mark by adding a few drops of distilled water.

Immediately before using, the alcohol was distilled and carefully brought to 25°C. before pipetting the required quantity. For comparative purposes an aqueous gliadin solution was prepared in the same way and also the corresponding water-alcohol mixtures. As contraction takes place upon diluting alcohol with water, the densities of the alcohol-water mixtures were measured with a picnometer, and with data of alcohol tables the percentages of alcohol by weight were calculated. (Column 5, Table I.)

The viscosity measurements were made in an Ostwald viscosimeter at a temperature of 25 ± 0.01°C., one hour after the dilutions had been made. Complete figures for one series of measurements are given in Table I and Figure 1.

Einstein deduced theoretically the following equation for the viscosity of colloidal solutions: $\frac{\eta_s}{\eta_0} = 1 + 5/2\ \phi$, where η_s is the viscosity of the total system, η_0 the viscosity of the intermicellar liquid, ϕ the volume of the particles as part of the total volume. This equation can only be used: (1) in case of a small value of ϕ, that is, in very dilute solutions, (2) when the particles are spherical and elastic; small in relation to diameter of the capillary, large in comparison with the molecules of the dispersion medium.

Calculating ϕ for emulsoids from viscosity data, it is found that this value is many times larger than it should be according to the calculation from the concentration of the dispersed substance. We found that ϕ of an acid gliadin solution of 0.25% at the maximum of viscosity has a value ± 15 times larger than would be expected. These excessively large values characteristic of emulsoids can be explained by assuming that the colloid particle binds a part of the dispersion medium. A sphere of orientated water molecules will be the result (hydration layer). Thus the value ϕ determined for emulsoids will give the volume of the particles with their hydration layer.

In colloidal problems it is important to determine the change of hydration of the particles (change of ϕ). We therefore calculated from measurements the relative viscosity of the gliadin in different alcohol media, (Column 4, Table I) by dividing the figures of Column 3 (the viscosity of the alcoholic protein solution) by those of Column 2 (the viscosity of the corresponding alcohol-water mixtures). Column 4 indicates the change of the value $1 + 5/2\ \phi$ in varying alcohol media.

In the tables and figures in this paper, we will indicate the viscosity of the protein solution in alcohol media with η_{S+A}, the viscosity of the alcohol-water mixtures, with η_A, the viscosity of a watery gliadin solution with η_W, and the relative viscosity in varying alcohol medium with $\frac{\eta_{S+A}}{\eta_A}$

TABLE I.

VISCOSITY (η_{s+A}) AND RELATIVE VISCOSITY $\frac{\eta_{s+A}}{\eta_A}$ OF A POSITIVE GLIADIN SOL IN VARYING ALCOHOL CONCENTRATIONS

	Alcohol	η_A	η_{s+A}	$\frac{\eta_{s+A}}{\eta_A}$	Alcohol by weight, %
Diluted to 50 cc.	0 cc.	.....	.....	1.092	.....
	10 cc.	1.784	1.959	1.098	14.10
	15 cc.	2.233	2.452	1.104	22.20
	20 cc.	2.583	2.873	1.112	31.17
	25 cc.	2.780	3.119	1.122	39.40
	30 cc.	2.838	3.197	1.127	48.70
	35 cc.	2.760	3.091	1.120	58.90
	40 cc.	2.555	2.805	1.098	69.05
	45 cc.	2.245	2.340	1.042	80.60

In considering Table I and Figure 1 we find: (1) The curves of both the alcohol-water mixtures and the alcohol protein solutions pass through a maximum at the same alcohol concentration, (2) The relative viscosity curve at first rises slowly, then faster, and reaches a maximum at the same concentration as the alcohol-water mixtures; after that it falls rapidly.

The form of the relative viscosity curve shows some similarity with the phenomena described by Bungenberg de Jong (1923) upon adding alcohol to agar solutions containing tannic acid. He found that while the viscosity of an agar solution falls rapidly with increasing alcohol concentration, an agar-tannic acid solution first shows a sharp rise in viscosity upon adding alcohol, reaches a maximum, and afterward falls rapidly.

The author explains this phenomenon by assuming that the adsorption medium will be changed from suitable to unsuitable by

adding alcohol. Assuming this to be correct, we may conclude in general that a curve of similar form means that on the surface of the colloidal particle a surface-active material has been absorbed. In our case it is improbable that tannic-acid derivatives should be responsible for this form of curve for the following reasons:

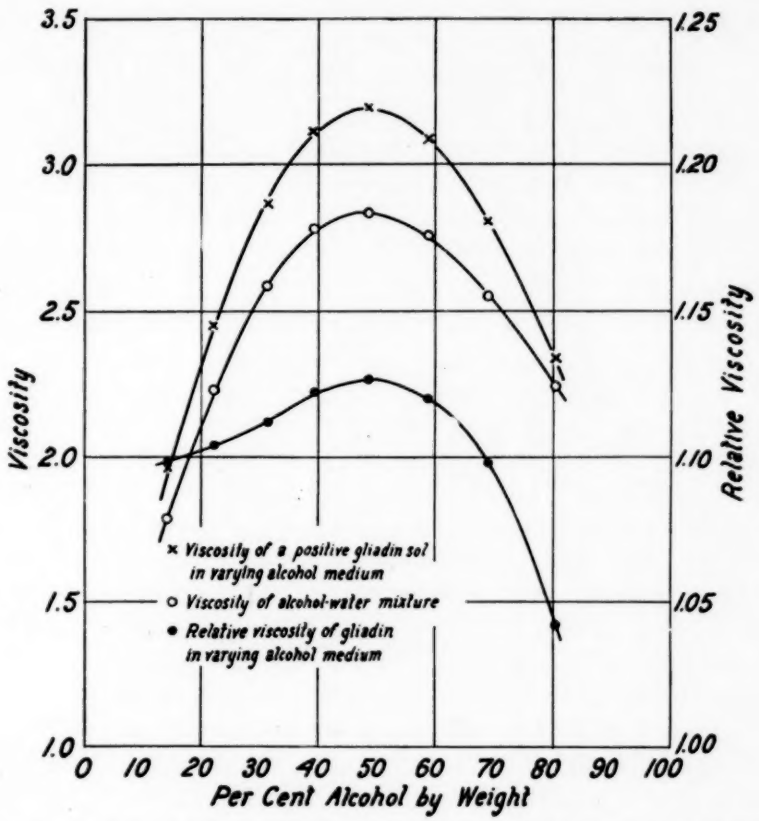

Fig. 1. Viscosity and relative viscosity of gliadin sols prepared with alcohol of varying concentration.

(1) No tannic acid can be detected in the preparation itself or in the aqueous residues of the preparations, (2) On the negative side of the isoelectric point the curve shows the same form as on the positive side, by adding alcohol. Tannic acid cannot be adsorbed on the negative side because of the forming of tannate. It is, however, possible that some other substance is adsorbed on the surface. In that case this substance must be firmly adsorbed on the acid as well as on the alkali side.

The increase in the relative viscosity of gliadin in varying alcohol concentrations expressed in percentage of the viscosity of the watery solutions minus one, indicates the percentage change in the hydration of the particles. In this paper we will call this factor percentage rise in relative viscosity. These values calculated from Table I are found in Table II.

TABLE II

CHANGE OF THE PERCENTAGE RISE OF THE RELATIVE VISCOSITY OF AN ACID GLIADIN SOL IN VARYING ALCOHOL CONCENTRATIONS

Alcohol by weight, %	Rise, %
14.06	8
22.20	13
31.17	22
39.40	33
48.70	38
58.83	30
69.05	7
80.56	—54

The increase of this value with increasing alcohol concentration, which reaches a maximum in 48% alcohol by weight, must not be entirely due to the increase in volume of the particles with water layer (ϕ), but the electric term, calculated by Smoluchowsky, may be subject to change. This term contains as variable factors ζ, the electro-kinetic potential, D the dielectric constant, K the conductivity, and r the radius of the particles.

It is certain that the dielectric constant of the medium changes with varying alcohol concentration, and coupled therewith, also ζ. Also the conductivity of the solution will be subject to change. Which part of the percentage rise in the relative viscosity at the maximum may be charged to the account of the electric factor is unknown, hence the exact part of the percentage rise due to the increase of size of the hydration layers is unknown.

That, however, in 48% alcohol by weight at this maximum the particle is in an extraordinary state, is shown by experiments of Dill and Alsberg (1925) and by Gottenberg and Alsberg (1927). The first two investigators found that the critical peptization of their gliadin preparations from different origins was lowest in solutions containing 60% alcohol by volume, whereas the latter investigators determined the effect of heat on the C. P. T. in different alcohol concentrations. The tables of Gottenberg and Alsberg show that the least fluctuation of the values of the C. P. T., plotted against time, took place in 60% alcohol by volume. We found that the stability of a gliadin solution at the maximum was increased,

as the decrease in the viscosity of this solution plotted against time, when compared with the water sol, was a considerably lower percentage.

We suggest that the increased stability of the gliadin solution must be found in a possible alcohol-water layer of the particles, because the maxima of the alcohol-sol viscosity and of the alcohol-water viscosity are found at about the same percentage of alcohol in solution. This indicates that in this concentration of alcohol something particular is going on; 46% alcohol by weight corresponds in molecular relation to one molecule of alcohol to three molecules of water. We therefore suppose that the maximum of the viscosity curve of alcohol-water mixtures is caused by formation of an alcohol hydrate. We assume that this hydrate formation also takes place in the water layer of the colloid particles, as from experiments on the lyotropic influence of univalent ions it follows that the particle in this alcohol concentration finds itself in a very special state (see later).

The decrease in the percentage rise in relative viscosity at a concentration of alcohol of more than 48% shows that a different process takes place. As mentioned before, this magnitude is proportional to the increase of ϕ, and we find consequently that the particles become smaller with increasing alcohol concentration. In other words, by increasing the alcohol concentration, water will be gradually removed from the alcohol-hydrate layer, as the medium must be in equilibrium with the surrounding liquid layer of the particle. Evidently this particle cannot exist with a pure alcohol layer and the particle has lost its protecting layer. With this decrease of viscosity is coupled a slow opalescing of the gliadin solution, until a clear blue solution is obtained at a concentration of about 80% of alcohol, which shows a strong Tyndall effect. On adding traces of electrolytes the solution became cloudy, and the protein slowly settled down. The low viscosity, the blue color, the Tyndall effect of this protein solution, and the sensibility to electrolytes, indicate that a gliadin with a more suspensoid character has been obtained, stable mainly on its charge.

Summing up, we find that alcohol up to a concentration of 48% by weight increases the protecting layer and therefore gives a stabilizing effect; at a higher percentage of alcohol in solution, the protecting layer is gradually removed from the particle.

This is in accordance with the following experiment: If the influence of changing the H-ion concentration on acid gliadin solu-

tions containing respectively 20%, 48%, and 75% alcohol by weight is compared, it appears that on adding equal quantities of dilute alkali to these solutions the mixtures containing 20% and 75% alcohol become cloudy, and that at the isoelectric point the protein settles down out of the solution, while the solution containing 48% alcohol remains clear. Again, it is evident that alcohol of 48% by weight has a stabilizing influence.

Influence of H-ion Concentration on the Viscosity of Alcoholic Positive Gliadin Solutions.

In further experimenting, two questions arose:

1. Does the total aspect of the curve (η — % alcohol) change upon changing the H-ion concentration of the gliadin sol?
2. How does the percentage rise in the relative viscosity in the maximum of the curve (η — % alcohol) change upon changing the H-ion concentration?

While the aqueous gliadin solution mentioned (6 cc. 0.1 HCl) did not show maximum viscosity, we now prepared a sol in the same way, but added so much HCl that the protein was just on the point of maximum viscosity in the η — pH diagram. We give below one of the tables of measurements (Table III).

TABLE III

INFLUENCE OF VARYING ALCOHOL CONCENTRATIONS ON THE VISCOSITY AND RELATIVE VISCOSITY OF AN ACID GLIADIN SOL WITH MAXIMAL HYDRATION

	Alcohol	η_A	η_{a+A}	$\frac{\eta_{a+A}}{\eta_A}$	Alcohol by weight, %
Diluted to 50 cc.	0 cc.	.....	.....	1.103	0.0
	10 cc.	1.781	1.977	1.107	14.0
	20 cc.	2.579	2.910	1.128	31.0
	25 cc.	2.782	3.161	1.136	39.5
	30 cc.	2.841	3.227	1.136	48.7
	35 cc.	2.762	3.109	1.125	58.8
	45 cc.	2.257	2.386	1.057	80.0

It is evident that the relative viscosity of this sol reaches its maximum at the same alcohol concentration as the relative viscosity curve mentioned in Table I, Column 4, irrespective of the experimental error. The shape of the two curves appears to be the same; consequently change of the H-ion concentration gives no change in the qualitative effect of the alcohol. The value of the percentage rise in viscosity with varying alcohol percentages is given in Table IV.

On comparing the readings of Tables II and IV it appears that the increase of the percentage rise for 48% alcohol by weight is

TABLE IV.

CHANGE OF THE PERCENTAGE RISE OF THE RELATIVE VISCOSITY OF AN ACID GLIADIN SOL WITH MAXIMAL HYDRATION IN VARYING ALCOHOL CONCENTRATIONS

Alcohol by weight, %	Rise, %
14.0	4
31.0	24
39.5	32
48.7	32
58.8	21
80.0	—45

dependent on the pH of the gliadin solution, that is, on the hydration of the particles.

In order to get a closer insight into the change of the percentage rise in the relative viscosity in a 48% alcohol medium upon changing the H-ion concentration (the medium where the particles acquire their maximum stability) we made the following experiment.

We started with a solution prepared by shaking 3 gm. of gliadin with 105 cc. distilled water and 5 cc. 0.1N HCl. Into a 25 cc. volumetric flask, containing 15 cc. alcohol and varying quantities of distilled water and hydrochloric acid, 2.5 cc. of this gliadin solution was pipetted. After bringing the solution to 25°C. in a thermostat, we filled the flask up to volume. In the same way the aqueous solutions were made with the same quantity of acid added that was added to the alcohol-gliadin solutions. We then measured the viscosity of these solutions and by aid of the viscosity of the corresponding alcohol-water mixtures, the value $\frac{\eta s + A}{\eta A}$ was calculated. Table V shows the quantities of acid added and the value.

TABLE V.

CHANGE IN VISCOSITY IN WATERY MEDIUM (η_s) OF A CLEAR GLIADIN SOL AND OF THE RELATIVE VISCOSITY $\left(\frac{\eta_{s+A}}{\eta_A}\right)$ IN ALCOHOL 48% BY WEIGHT, BY ADDITION OF VARYING QUANTITIES OF HYDROCHLORIC ACID

cc. 0.01N Hcl	0	1	2	5	10	20	50
η_s'	1.077	1.104	1.103	1.085	1.070	1.056	1.039*
$\eta_{s'+A}$	3.171	3.229	3.212	3.142	3.091	3.057	3.030
η_A	2.833	2.833	2.833	2.833	2.833	2.833	2.833
$\frac{\eta_{s+A}}{\eta_A}$	1.119	1.140	1.134	1.109	1.091	1.079	1.070
Rise, %	54	35	30	29	30	41	80

* Cloudy

The values in column marked η_s represent the change in viscosity of the watery gliadin solution with changing acidity. It reaches a maximum when the charge on the particle passes through the maximum; afterward the viscosity decreases in consequence of the discharging effect of the negative ions. The value $\frac{\eta_{S\ A}}{\eta_A}$ shows a similar curve, which, however, does not run parallel with the water curve.

This deviation cannot be put down to the difference in H-ion concentration of the alcohol and water medium, as it appears from Figure 2 that the maxima of the viscosity curves of alcohol sol and water sol occur with practically the same quantities of acid added. It might be expected that these curves would run parallel, assuming that alcohol at different pH exercises the same action on the particles. As this is not the case, it is evident that the influence of alcohol on ϕ is dependent on the H-ion concentration. The percentage rise in the relative viscosity decreases until the maximum charge is attained. At this point it should be $\pm$ 33% interpolated from the data of the curve. On increasing the acidity of the solution beyond this point, the percentage rise still decreases slightly, and afterward increases abruptly. It can be observed with the naked eye that the action of alcohol is different at varying pH, because the solution in watery medium (marked *), was cloudy, while the alcohol solution was entirely clear.

It appeared of interest to investigate more closely the action of 48% alcohol by weight on very milky gliadin solutions, which showed under the microscope very marked forming of liquid drops. Gliadin solutions were made with $\pm$3 gm. gliadin but with different quantities of acid not sufficient for complete peptization. While the preparation of clear acid-gliadin solutions was from day to day perfectly reproducible, we have not succeeded in reproducing the separated sols identically, because small differences in time of shakinging, traces of electrolyes, and small quantities of undissolved protein had marked influence on the viscosity. We may only compare the figures of those experiments which resulted in preparations having the same viscosity. The mode of operation was the same as in former experiments. Table VI and Figure 2 give the series C. These sols contain in 25cc., 2.5 cc. separated protein solution and 15 cc, alcohol. The solutions C_1 and C_2 were made in the same way, but differed slightly, owing to the reason mentioned above. The quantity of added HCl is stated in millimols per liter.

TABLE VI

THE INFLUENCE OF VARYING QUANTITIES OF HCl ON THE VISCOSITY OF UNMIXED GLIADIN SOLS AND THEIR RELATIVE VISCOSITIES IN ALCOHOL 48% BY WEIGHT

	C 1		2.5 cc. Sol diluted to 25 cc.					
	3.056	3.127	3.186	3.209	3.185	3.143	3.091	η_{s+A}
	45223	45223	45223	45223	45223	45223	45223	$\log \eta_A$
15 cc. alcohol per 25 cc.	1.078	1.104	1.125	1.133	1.124	1.110	1.091	$\frac{\eta_{s+A}}{\eta_A}$
	1.039	1.075	1.093	1.101	1.096	1.085	1.071	η_s
	100 %	39 %	34 %	32 %	30 %	29 %	28 %	Rise, %
	0	0.2	0.4	0.8	1.2	2.0	4.0	m. mols. HCl per L

	C 2		2.5 cc. Sol diluted to 25 cc.			
	3.006	3.132	3.209	3.178	3.137	η_{s+A}
	45223	45223	45223	45223	45223	$\log \eta_A$
15 cc. alcohol per 25 cc.	1.061	1.106	1.133	1.122	1.107	$\frac{\eta_{s+A}}{\eta_A}$
	1.028	1.075	1.099	1.091	1.083	η_s
	116	40	34	31	29	Rise, %
	0	0.4	0.8	1.2	2.0	m. mols. HCl per L

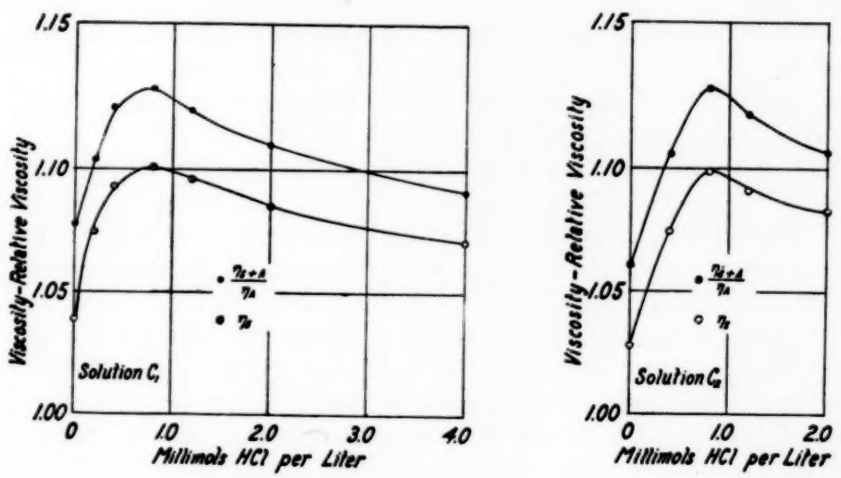

Fig. 2. Change in viscosity and relative viscosity of sols (C_1 and C_2, Table VI) at varying concentrations of HCl.

Measurements were made after 24 hours, because the separated solutions were not in equilibrium before that time. The clear watery solutions near the maximum of viscosity were in equilibrium within an hour.

As in former determinations, we find here, by adding acid, an initial decrease of the percentage rise in the relative viscosity. In the case of separated sols this rise has a very high value. At the maximum of charge of gliadin-water solutions, we find on adding alcohol up to 48% by weight, a rise of 32% for C_1 and of 34% for C_2. From earlier determinations we found about 32%. The average of these and other determinations we find to be about 33%.

We further investigated whether this percentage rise in the maximum was strongly dependent on the concentration of the protein in solution. We started with solutions containing the double quantity of protein (B), i. e. 5 cc. protein solution in 25 cc. The method was the same as in previous experiments. As appears from Table VII, Figure 3, at the maximum there is a percentage rise of 35% and 34%, which is in accordance with earlier determinations (C), considering that deviations of 2% may occur. (Table VII and Figure 3.)

We conclude that, irrespective of experimental error, the percentage rise in the relative viscosity in 48% alcohol by weight, at the maximum charge of the particles, is independent of the protein concentration of those solutions, provided these solutions are very dilute.

At this maximum we find in alcoholic medium (48%) for this gliadin preparation a rise of $\pm$ 33% of the original viscosity of the hydrosol. In other words, a particle at maximal charge increases in volume in 48% alcohol by one-third (not considering the change in value of the electric term of the Einstein-Smoluchowsky formula) which increase is due to the forming of a possible alcohol-hydrate layer.

In order to obtain a general view, we now plot the percentages of rise in relative viscosity against the corresponding viscosity of the water sol, and from each series of experiments we get a curve, which, in general, has the same shape but which differs in height (Fig. 4). We see that to every viscosity in water medium belong two values of percentage rise in relative viscosity in 48% alcohol medium.

Owing to the lack of data in the literature, we cannot conclude that these two percentage rises at the same viscosity indicate dif-

TABLE VII.

INFLUENCE OF VARYING QUANTITITES OF HCl ON THE VISCOSITY OF SEPARATED GLIADIN SOLS OF DOUBLE CONCENTRATION AND THEIR RELATIVE VISCOSITIES IN ALCOHOL 48% BY WEIGHT

B 2	5 cc. Sol diluted to 25 cc.					
15 cc. alcohol per 25 cc.	3.254	3.501	3.516	3.483	3.342	$\frac{\eta_{s+A}}{\eta_w}$
	45223	45223	45223	45223	45223	$\log \eta_A$
	1.149	1.236	1.241	1.229	1.215	$\frac{\eta_{s+A}}{\eta_A}$
	1.093	1.164	1.178	1.176	1.169	η_s
	60	38	35	30	28	Rise, %
	0	0.8	1.2	1.6	2.4	m. mols. HCl per L

B 3	5 cc. Sol diluted to 25 cc.				
15 cc. alcohol per 25 cc.	3.282	3.411	3.477	3.505	$\frac{\eta_{s+A}}{\eta_w}$
	45223	45223	45223	45223	$\log \eta_A$
	1.159	1.204	1.227	1.237	$\frac{\eta_{s+A}}{\eta_A}$
	1.101	1.151	1.170	1.177	η_s
	57	35	34	34	Rise, %
	0	0.4	0.8	1.2	m. mols. HCl per L

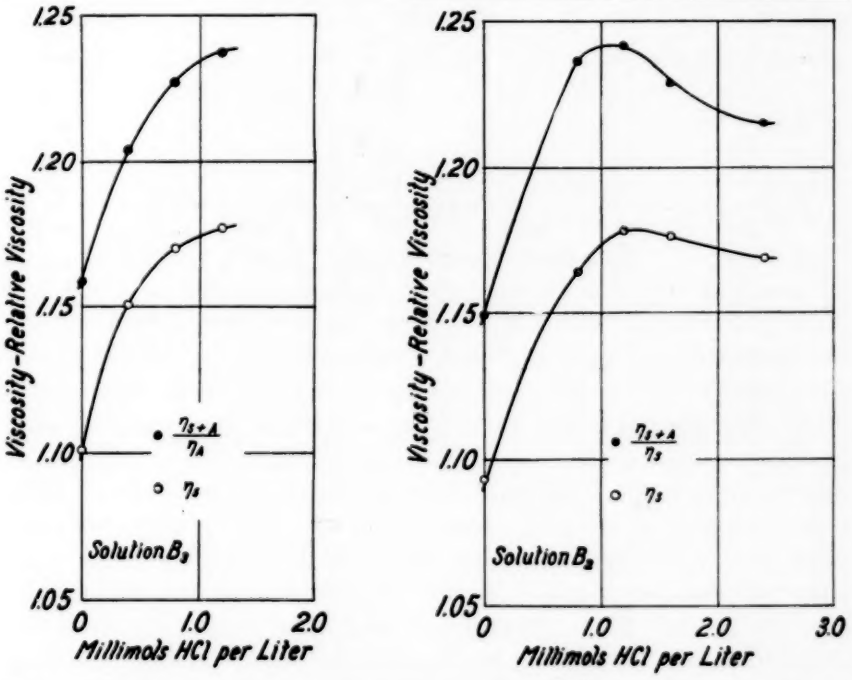

Fig. 3. Change in viscosity (η_s) and relative viscosity ($\frac{\eta_{s+A}}{\eta_A}$) of gliadin sols ($B_2$ and B_3 Table VII) at varying concentrations of HCl.

ferences in ϕ, and also in the Smoluchowsky term (which in that case must compensate each other) or whether ϕ is equal and the cause of these differences is the varying factors in the electric term, while the total value of this term in both cases must be the same. Neither can we explain the rapid decline of a part of this curve immediately after the maximum.

We find that on both sides of the maximum the solutions show a larger percentage rise and that these indicate different areas of separation; the rise being greater in the same measure as the separation proceeds, as shown microscopically, but that clear solutions, which do not show liquid drops microscopically, nevertheless have a larger percentage rise than at the maximum.

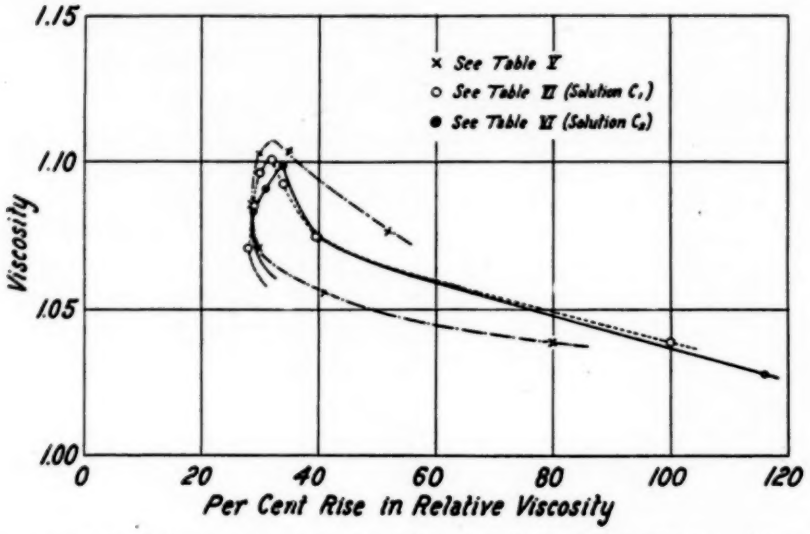

Fig. 4. Relation between per cent rise in relative viscosity in alcohol 48% by weight and the viscosity of aqueous gliadin sols prepared at various concentrations of acid.

It is desirable to direct our attention to what can be observed through the microscope upon adding alcohol to the separated solutions. We see the liquid drops (which for this purpose have been carefully dyed with fuchsin) swell when adding alcohol; the outlines become vague, and at length the solution becomes optically void. In the first instance, alcohol has a dispersing effect. We assume that this effect must be the cause of the large percentage rises in relative viscosity in the separated solutions. When the particle has reached its maximal charge, the rise in viscosity amounts to about 33% in 48% alcohol solution. In the separated solutions are larger complexes of protein particles. The larger per-

centage rises, found in these solutions, must be attributed to (1) the dispersing effect of alcohol on these complexes; and (2) the effect of enlarging the protecting layer of the single particle.

As the shapes of the curves (Fig. 4) are perfectly regular for both separation areas, it follows that separation is a *continuous* process, in the microscopically visible area as well as in the amicroscopical area. Therefore the separation gradually decreases from the isoelectric point up to the maximum of charge. Afterward, with decreasing charge, the separation gradually increases, irrespective of the sudden drop in the curve after the maximum.

Influence of Alcohol on the Relative Viscosity of a Negative Gliadin Solution

On the negative side of the isoelectric point only a few determinations were made. By adding a quantity of hydroxide too small for complete peptization, separation resulted; with larger quantities of hydroxide a clear solution can be obtained. The method of preparation of the gliadin solutions was the same as mentioned for the measurements on the acid side, except that the water was boiled free of CO_2, and utmost care was taken that no CO_2 should penetrate into the solutions. Therefore in the viscosity determinations the viscosimeters were fitted with soda-lime tubes.

The phenomena here found by adding alcohol were the same as on the acid side. With the first quantities of alcohol added, the cloudy separated solutions became opaque, and, by further addition, clear. The maximum in viscosity was found at a slightly lower concentration of alcohol than on the acid side. By increasing the alcohol concentration after this maximum, a decrease of viscosity took place. With this decrease in viscosity was coupled, in the homogeneous gliadin solutions, a blue opalescing of the liquid, while in the definitely separated solutions, increasing turbidity resulted. The more the solution was separated, the larger became the percentage rise in viscosity at the maximum of the η—alcohol curve.

Table VIII and Fig. 5 give the results of one of our measurements.

Summing up, we find on the alkali side that alcohol has first a dispersing effect, coupled with a stabilizing effect, which reaches its maximum in $\pm$ 50% alcohol by volume. Afterward dehydration takes place, and the gliadin passes gradually into a suspensoid.

A fine example of the increased stability at the maximum can

TABLE VIII.

INFLUENCE OF ALCOHOL ON THE VISCOSITY η_{s+A}, AND THE RELATIVE VISCOSITY OF A NEGATIVE GLIADIN SOLUTION

Alcohol by volume, %	0	20	30	40	50	60	70	80	90
Appearance	——	op.	l.op.	clear	cl.	cl.	cl.	l.op.	op.
η_{s+A}	.....	1.856	2.323	2.708	2.921	2.981	2.891	2.644	2.289
η_A	.....	1.784	2.223	2.583	2.780	2.838	2.760	2.555	2.245
$\frac{\eta_{s+A}}{\eta_A}$	1.034	1.040	1.045	1.049	1.051	1.050	1.047	1.035	1.019

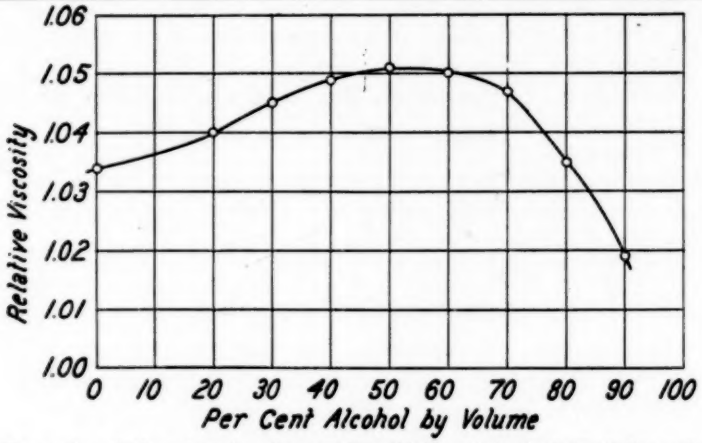

Fig. 5. Change in relative viscosity of a negative gliadin sol in alcohol solutions of varying concentration.

be demonstrated with alkali-gliadin sol, with varying concentrations of alcohol.

By standing open, with the same dimension of contact surface to the air, the solutions with the smallest and largest quantities of alcohol became turbid in a short time. With 40% and 70% alcohol by volume, after some time a slight turbidity developed. After standing overnight, only the solution at the maximum was absolutely clear, with 60% alcohol slightly opaque, and the others milky in different degrees.

The action of CO_2 in the air is as follows: It combines with the alkali with formation of carbonates. The result is a decrease of the H-ion concentration in the direction of the isoelectric point.

Separation Phenomena

At this point it is desirable to examine the phenomena of separation more in detail.[1] Before proceeding to a possible explanation

[1] At the same time (not yet published) H. R. Kruyt and H. G. Bungenberg de Jong found that the separation phenomena can occur in all emulsoids under special conditions. They call these phenomena coacervation.

of the facts, we must lay stress on the following: (1) As the globules magnify by decrease of their charge, they are never seen to collide or fuse, unless in the neighborhood of the isoelectric point. (2) The magnitude of the drops equally charged is for some time variable within wide limits. It appears that by adding a precipitating electrolyte to a gliadin solution, slowly or quickly, entirely different states of cloudiness can be called forth. The solution obtained by quick addition are clearer than the others, and the microscopic image shows that by adding slowly the magnitude of the drops is more uniform than by adding quickly. In the latter case we find in the midst of many very small ones, drops 4 to 6 times their diameter. The viscosity of the two solutions was the same within experimental error, which indicates that the total volume of the particle in both solutions was the same. (3) Upon heating separated solutions till they are clear, and subsequent slow or quick cooling to room temperature, the magnitude of the drops may be largely varied, but in this case the viscosity also varies. (4) At the isoelectric point protein strings are formed in the foam. These observations can be brought under the following train of thought: By decreasing the charge of the colloid particles, centers of separation will be formed. The magnifying of the centers cannot be caused by collision, because they are charged positively and are mutually repulsive. If we take away a part of the hydration of a colloid particle lying in the liquid intermediate between the centers by means of a decrease of charge, this particle may be considered as a submicroscopical center. The centers are all positively charged and are mutually repulsive unless there is a force stronger than the repulsive action. This force we take to be the difference of surface tension between two particles of different magnitude. If this force is greater than the repulsive action, the smaller particles will join the larger ones. We assume, therefore, that the difference in surface tension between drops of different magnitude is the cause of the magnifying of the globules. This explains why, after quickly adding electrolyte, the solution gradually reaches equilibrium and in that state the drops are of the same order of magnitude and consequently must have the same surface tension. Upon reaching the isoelectric point in consequence of the discharging of the particles, the repulsive force should have disappeared, and the drops will stick together. We must therefore assume a protein droplet to consist ultimately of partially dehydrated gliadin particles as the continuous phase forming a reticulum with water

in the interstices as a discontinuous phase. This also explains why, by increasing charge and hydration of each of the gliadin particles, a decrease of magnitude in the protein drops results. The surface layer of the protein drops will be charged through change of medium. The repulsive force, caused by increase of charge, added to the pressure caused by the magnification of the water layer of those particles, will become greater than the surface tension. The particles will go in colloidal solution, until equilibrium is reached.

The next question is: Which substances will flocculate and which will separate upon discharging? In principle, in separation the liquid nature of the sediment predominates, and in flocculation the solid phase. We must therefore search for the difference between substances or combinations thereof that separate or flocculate, in the water-binding capacity at the isoelectric point; viz., in the hydration independent of charge. Substances having great hydration independent of charge, will at the isoelectric point neither flocculate nor separate, e. g., gelatin and albumin. The other extreme will be found in protein having a small isoelectric hydration, e. g., casein and, in greater measure, glutenin. As an intermediate class, we may take gliadin and perhaps the other prolamins. These substances have a medium isoelectric hydration, which, however, is not of sufficient magnitude to keep them discharged in solution at the isoelectric point. The latter will show separation.

This theory, namely, that the isoelectric hydration (i. e., the quantity of water bound by the protein in uncharged condition) and not the hydration dependent on charge, is solely responsible for the phenomena of separation, flocculation, and remaining in solution by the discharging of different protein sols, is founded on the following facts:

(1) It is possible by addition of foreign substances, to increase the isoelectric hydration of casein and glutenin in such a way that these substances, which will otherwise flocculate, are able to separate. (2) Albumin that in ordinary circumstances remains in solution at the isoelectric point, can be separated by certain physical manipulations or the addition of various substances. (3) In alcohol 48% by weight the behavior of gliadin has completely changed. By the discharging of an acid alcoholic gliadin sol, the gliadin remains completely in solution. The reason is that this medium increases the isoelectric solvation of the gliadin. In this medium gliadin can-

not be salted out by means of electrolytes, even in high concentration (4 mols).

The isoelectric hydration of the gliadin has a medium value; it is impossible to get flocculation in watery solutions, but the result is always separation unless a substance is added that alters the isoelectric hydration. By discharging the colloid particles, the hydration caused by charge will be removed; but the small concentration of electrolyte necessary for the discharging will not sufficiently alter the isoelectric hydration of the gliadin, with the exception of the complex salts (phosphotungstic acid and others). These salts will first discharge the particle and afterward will be adsorbed on the surface of the particle and will remove the isoelectric hydration.

By discharging the gliadin in dehydrating media, other phenomena can be observed; e.g., an acid alcoholic gliadin sol will flocculate by decrease of charge, when the alcohol concentration in the solution is about 80%. In this medium the gliadin particle gets a more suspensoid character by losing its protecting layer. By discharging the particles, the solid nature will predominate and flocculation will result.

Following papers will deal with the lyotropic influence of different electrolytes on acid gliadin sols in water, alcohol, and acetone media.

Summary

1. The influence of varying alcohol concentration on positive and negative gliadin sols was studied.
2. Up to a concentration of alcohol 48% by weight an increase in the relative viscosity takes place. In higher alcohol concentrations a rapid fall in these values occurs with both positive and negative gliadin.
3. This maximum in the relative viscosity is supposed to be caused by the forming of an alcohol hydrate layer round the particles.
4. The fall in the relative viscosity in higher alcohol concentration is caused by a gradual removing of this layer.
5. The influence of changing H-ion concentration on the maximum in 48% alcohol by weight was studied by means of the percentage rise in relative viscosity.
6. At both sides of the maximum hydration of the aqueous sol, an area of separation was found, as indicated by very large values of the percentage rise in the relative viscosity.

7. The gradual decrease of the percentage rise in relative viscosity with changing H-ion concentration indicates that separation is a gradual process.
8. A theory on separation phenomena is given.
9. Whether an emulsoid will flocculate, unmix, or remain in solution by discharging, is only dependent on the isoelectric hydration.
10. It is possible to change the isoelectric hydration so that every protein can be separated under special conditions.

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THE EFFECT OF ACID POTASSIUM TARTRATE AS AN INGREDIENT IN ANGEL CAKE[1]

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(Received for publication January 17, 1930)

Angel cake in which acid potassium tartrate is incorporated is white and fine-grained, whereas without it the cake is yellow and coarse-grained. Observations of these differences led to an investigation to ascertain the cause.

Experimental

The cakes used in this investigation were made according to the following formula and method of procedure:

Sugar	150 grams
Flour	50 grams
Egg whites	130 grams
Salt	1 gram
Vanilla	2 cc.
Acid	Variable

The egg white, salt, and acid potassium tartrate were placed in the 3-quart bowl of the Hobart mixer and beaten approximately 2¾ minutes at high speed with the flat open paddle, the time of beating varying slightly with age of egg and amount of acid used. Thirty-five grams of sugar was sifted over the beaten mixture and gently folded in with an egg whip, and this was followed by a second addition of 35 grams of sugar incorporated in the same manner as the first addition. The remaining portion of sugar was sifted with the flour, and the sugar-flour mixture was divided into three portions, each of which was sifted over the mixture and gently folded into it. Lastly the vanilla was added and the folding continued for 30 seconds. At the beginning of the experiment the temperature of all the ingredients was 75°F. and the work was done in a room maintained at that temperature. The cakes were baked in tube pans of 16½ centimeters in diameter and 7½ centimeters in height, at 310°F.

[1] Published with the approval of the Director as Paper No. 937, Journal Series, Minnesota Agricultural Experiment Station.

[2] This investigation was started while the senior author was a student at the University of Minnesota, and completed in the Bureau of Dairy Industry.

Hydrogen-ion concentration determinations were made upon the products with the quinhydrone electrode. Color determinations were made in terms of the Munsell system of notation. The system is described by Cleland (1921) and a method for its application by Nickerson (1929).

Effect of Variation in the Quantity of Acid Potassium Tartrate

Four cakes were made in which 0.0, 0.4, 0.8, and 1.2 grams of acid potassium tartrate were incorporated. The data obtained from this experiment are recorded in Table I. Acid potassium tartrate has the effect of an acid. With the above quantity of ingredients an increase of each 0.4 gram of acid potassium tartrate resulted in a change of H-ion concentration represented by about 0.4 pH unit.

TABLE I

EFFECT OF QUANTITY OF ACID POTASSIUM TARTRATE UPON THE HYDROGEN-ION CONCENTRATION AND THE COLOR OF ANGEL CAKE

Cake Designation	Quantity of Acid Potassium Tartrate	Hydrogen-ion Concentration of Cake	Munsell Notation		
			Hue	Brilliance	Chroma
	grams	pH.	Scale of 100	Scale of 10	Scale of 10
A	0.0	8.3	14.40	8.48	4.71
B	0.4	7.8	14.45	8.80	4.16
C	0.8	7.2	14.52	8.99	3.72
D	1.2	6.6	14.56	9.15	3.46

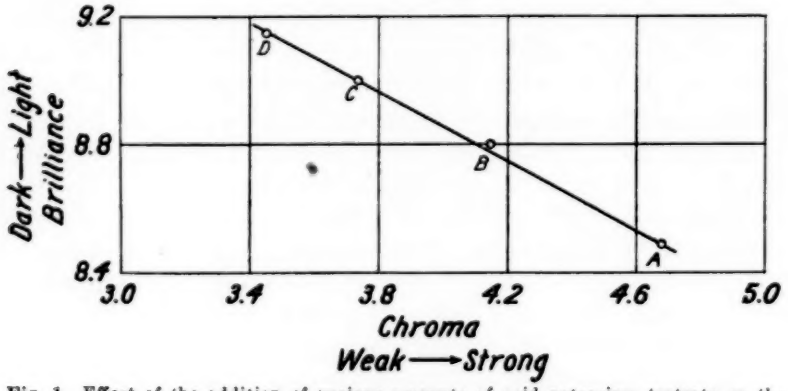

Fig. 1. Effect of the addition of various amounts of acid potassium tartrate on the hydrogen ion concentration as pH, and on the color in terms of brilliance and chroma, of angel cake.

The effect of variation in acid potassium tartrate on color in terms of hue is very slight, as is evident from Table I. With each increase in this ingredient the cake became more nearly white, as expressed by brilliance. The effect is that of a direct relationship.

Chroma or intensity is also affected by becoming considerably weaker. The data are reproduced graphically in Figure 1.

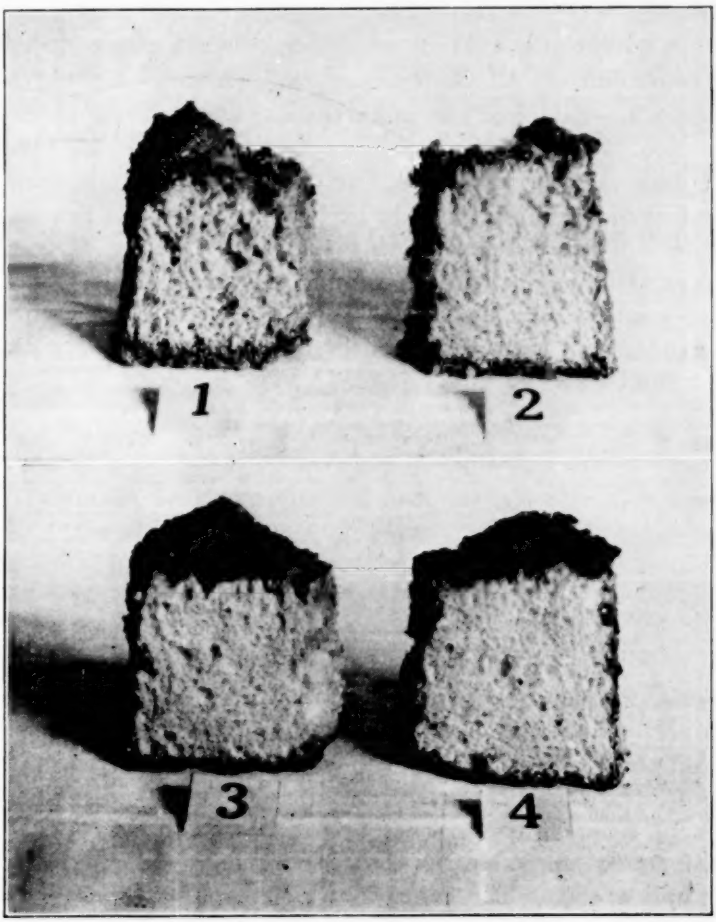

Fig. 3. Effect of varying amounts of acid potassium tartrate on grain of angel cake.
No. 1=0.00 grams
No. 2=0.40 grams
No. 3=0.80 grams
No. 4=1.20 grams

When no acid potassium tartrate was used the cells were large and thick-walled; with increase in acid potassium there was a decrease in the size of the cells and the thickness of the walls. Figure 2 illustrates four cakes in which quantity of acid potassium tartrate was the variant.

Cause of Change in Color and Grain of Angel Cake Due to Use of Acid Potassium Tartarate

The following experiments were made to determine whether the changes in color and grain of angel cake were due principally to a change in H-ion concentration: (1) effect of the use of tartrate, (2) of various acids on the H-ion concentration and color of angel cake, and (3) effect of age of egg white.

Series 1.—Effect of use of tartrate. In this series three cakes were made. The first contained no tartrate, the second contained 1.20 grams of acid potassium tartrate, and the third contained 1.34 grams of sodium potassium tartrate. The results are recorded in Table II. The cake containing sodium potassium tartrate was similar in H-ion concentration and in color and grain to that which did not contain any tartrate. From this it is evident that the changes that occur as a result of the use of acid potassium tartrate are not due to the tartrate.

TABLE II

EFFECT OF TARTRATE UPON THE HYDROGEN-ION CONCENTRATION AND THE COLOR OF ANGEL CAKE

Kind of Tartrate	Quantity of Tartrate	H-ion concentration	Munsell Notation		
			Hue	Brilliance	Chroma
	grams	pH.	Scale of 100	Scale of 10	Scale of 10
None.....	0.00	8.20	14.40	8.48	4.71
Acid potassium	1.20	6.60	14.56	9.15	3.46
Sodium potassium	1.34	8.05	14.41	8.33	4.75

Series 2.—Effect of various acids. Citric, malic, and tartaric, which are white, soluble, and non-poisonous acids, were used in place of potassium tartrate. Preliminary work showed that 0.48 gram of citric acid, 0.50 gram of malic acid, and 0.53 gram of tartaric acid would effect a change in H-ion concentration in the above formula equivalent to that produced by 1.20 grams of acid potassium tartrate. The cakes in which the four acids were used

TABLE III

EFFECT OF ACID POTASSIUM TARTRATE AND OTHER SOLUBLE ORGANIC ACIDS UPON THE HYDROGEN-ION CONCENTRATION AND COLOR OF ANGEL CAKE

Kind of of Acid	Quantity of Acid	H-ion Concentration	Munsell Notation		
			Hue	Brilliance	Chroma
	grams	pH.	Scale of 100	Scale of 10	Scale of 10
No acid	0.00	8.20	14.40	8.48	4.71
Potassium tartrate	1.20	6.62	15.00	9.00	3.17
Citric	0.48	6.58	15.00	9.11	3.11
Malic	0.50	6.59	14.52	9.07	3.12
Tartaric	0.53	6.58	15.00	9.11	3.20

were very similar in color, as is evident from the data recorded in Table III. From these data it would seem that the effect of the use of acid potassium tartrate in angel cake is that of its acidity.

Series 3.—Effect of age of egg white.—Sharp and Powell (1927) have shown that egg white, as a result of the effect of aging, may vary in H-ion concentration from 7.6 to 9.5 expressed as pH. To establish further that the changes in properties of angel cake that result from the use of acid potassium tartrate are due to a change in H-ion concentration, two cakes were made, one with fresh eggs and the other with eggs which had been in storage six weeks. There was a difference in the H-ion concentration of the two cakes and a noticeable difference in their color. The results of this series are recorded in Table IV.[3]

TABLE IV

EFFECT OF AGE OF EGG WHITE UPON THE HYDROGEN-ION CONCENTRATION AND COLOR OF ANGEL CAKE

Age of Egg White	H-ion Concentration (of Cake)	Munsell Notation		
		Hue	Brilliance	Chroma
	pH.	Scale of 100	Scale of 10	Scale of 10
One day	6.41	14.55	9.20	2.96
Six weeks	6.70	14.45	9.03	3.67

Experimental data are given which show that the tartrate radical by itself will not give the changes in color and grain in angel cake which result from the use of acid potassium tartrate and that other acids tested produce the same effect. It would seem that the changes in these properties are due largely to the acidity of acid potassium tartrate which causes a change in H-ion concentration of the medium.

SUMMARY

1. Angel cake made with acid potassium tartrate as a part of the ingredients is a fine-grained, white product, while without it the cake is yellow and coarse-grained. Use of acid potassium tartrate causes an increase in the H-ion concentration of the cake. The effect of increase of acid potassium tartrate on color in terms of hue is very slight, whereas there is a marked change in terms of brilliance and chroma.

2. Sodium potassium tartrate gives effects no different in color and grain from the control when substituted for acid potas-

[3] The egg whites used in procuring data for Tables I, II, and III were handled in a manner to insure uniform H-ion concentration of the egg whites for each experiment.

sium tartrate, consequently the tartrate radical may not be a factor in the effects produced with acid potassium tartrate.

3. Citric, malic, and tartaric acids used in place of acid potassium tartrate to change the H-ion concentration of the cake have the same effects on the color and the grain of the cake as acid potassium tartrate.

4. Egg whites which vary in H-ion concentration produce cakes which differ in color; the higher the H-ion concentration of the eggs and the resulting cake, the lighter the color of the cake.

5. It is concluded that the change in color and grain of angel cake resulting from the use of acid potassium tartrate is due largely to acidity.

ACKNOWLEDGMENT

The authors wish to acknowledge the assistance given by Dorothy Nickerson of the Bureau of Agricultural Economics, U. S. Department of Agriculture, in making the color determinations.

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FAT: ITS ESTIMATION IN WHEATEN PRODUCTS

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(Received for publication, October 2, 1929.)

INTRODUCTION

In an earlier paper by Herd (1927) the direct ether extraction by the Soxhlet method was discussed and it was shown that the heating of flour to remove the last traces of moisture resulted in a smaller quantity of fat being extracted by the solvent. Further, there was a progressive change in the refractive index and other properties of the fat, if this were heated at 98°C for a number of hours; this change did not appear to be an oxidation.

The present paper is an outcome of a criticism by Denham (private communication) that this change in the properties of the extracted material might probably be due to the retention in the fat of traces of water and solvent, which are gradually and slowly removed as heating progresses.

While this point was being investigated, the authors had occasion to determine the fat content of bread; the direct ether extraction gives practically a blank result, possibly due to the change in solubility previously noted, and a hydrolysis method has to be employed. Using one such method, it was found that the yield of fat was greater than the corresponding ether-extracted material in the flour from which the bread was made. A comparison, therefore, of the hydrolysis methods with the direct methods was undertaken and is here reported. Cormack (1926) reported differences in the amount of fat extracted by ether before and after peptic digestion.

The Association of Official Agricultural Chemists (Book of Methods, 1925) determined the crude fat officially by the direct extraction, but since then (1928) the Association has adopted the acid hydrolysis method as its official method for the estimation of fat in flour and baked goods.[1]

EXPERIMENTAL

The Effect of Solvent on the Constants of Wheat Fat

Throughout this work three representative samples of mill products were used. They are given in Table I with the moisture content at the time of removal from the mill.

[1] J. Assoc. Official Agr. Chem. 11:37.

TABLE I

Sample	Moisture %
Patent Flour (Top 50%)	14.86
Bran	14.26
Germ	14.02

Investigating the point raised by Denham, these products were extracted in the first place with commercial ether (undried); the ether was removed by a fan and then the fat was placed in a vacuum desiccator. Samples were removed after 24 and 48 hours and the constants determined; drying was then continued for 18 hours at 98°C and observations again made. These results are given in Table II. The refractive indices were obtained in an Abbé refractometer at 20°C and the bromine values by the method of Toms (1928). This is a very simple method and appears to give satisfactory results. Using flour fat, a quantity of 0.2 to 0.3 gm., gave a convenient increase in weight, and 20 minutes was sufficient time for complete absorption of the bromine. The iodine values were calculated from these bromine figures.

TABLE II

THE EFFECT OF DRYING ON THE PHYSICAL CONSTANTS OF FAT EXTRACTED BY COMMERCIAL ETHYL ETHER FROM MILL STOCKS

	Air Dried		After 24 hours in Desiccator			After 48 hours in Desiccator			After 18 hours at 98°C.		
Sample	N_D^{20}	Bromine Value	N_D^{20}	Bromine Value	Iodine Value	N_D^{20}	Bromine Value	Iodine Value	N_D^{20}	Bromine Value	Iodine Value
Approx.:											
Flour	1.4700	....	1.4830	66.6	105.76	1.4830	67.0	106.40	1.4890	57.9	91.95
Bran	1.4700	....	1.4800	68.0	108.00	1.4800	70.0	111.16	1.4820	68.0	108.00
Germ.	1.4700	....	1.4790	81.8	129.90	1.4790	81.3	129.10	1.4800	73.3	116.40

These constants appear to reach a definite value in the desiccator but to change on heating at 98°C. The fat extracted from the germ was more mobile than that from either the flour or the bran: all darkened in colour on heating, but the germ oil remained more mobile throughout.

Second portions of these products were then extracted in the Soxhlet apparatus with petroleum ether (boiling point 40°C. to 60°C.); this was chosen because it is immiscible with water and it is recommended by the English Board of Agriculture & Fisheries (1928) for the estimation of oil in feeding stuffs. Similar observations were made with the extracts thus obtained and the results are given in Table III.

TABLE III

THE EFFECT OF DRYING ON THE PHYSICAL CONSTANTS OF FAT EXTRACTED BY PETROLEUM ETHER (B.p.40°C. TO 60°C.) FROM MILL STOCKS

	24 hours in Desiccator		48 hours in Desiccator	12 hours in 98°C.	36 hours at 98°C.	
Sample	N_D^{20}	Bromine Value	N_D^{20}	N_D^{20}	N_D^{20}	Bromine Value
Flour	1.4830	67.5	1.4830	1.4870	1.4890	58.5
Bran	1.4810	70.0	1.4810	1.4810	1.4810	70.0
Germ	1.4800	81.5	1.4790	1.4852	1.4880	61.5

In the fats extracted from flour and germ, results are parallel to those in ethyl ether, namely a change in property on heating at 98°C. The fat from the bran in this case showed no such change. On heating all the fats darkened in colour.

On separate portions of these samples, the quantity of matter extractable by ethyl ether and petroleum ether was estimated by the method of Herd (1927) and weighed under varying conditions of drying, corresponding to the conditions employed above. These figures are given in Table IV.

TABLE IV

THE EFFECT OF DRYING ON QUANTITY OF FAT EXTRACTED BY ETHYL ETHER AND PETROLEUM ETHER FROM MILL STOCKS

	24 hours in Desiccator		48 hours in Desiccator		2 hours at 98°C.		15 hours at 98°C.		21 hours at 98°C.	
Sample	Ethyl Ether	Petroleum Ether	Ethyl Ether	Petroleum Ether	Ethyl Ether	Petroleum Ether	Ethyl Ether	Petroleum Ether	Ethyl Ether	Petroleum Ether
	%	%	%	%	%	%	%	%	%	%
Flour	1.38	1.21	1.37	1.19	1.20	1.16	1.21	1.10	1.20	1.10
Bran	4.59	4.32	4.58	4.29	4.35	4.18	4.35	4.19	4.34	4.19
Germ	8.40	7.87	8.40	7.85	8.26	7.82	8.26	7.75	8.26	7.75
	—— reduced to dry basis ——									

All results are the averages of satisfactory duplicates.

The quantity extracted by petroleum ether is in all cases lower than that extracted by ethyl ether. The weights are practically constant after 24 hours in the vacuum desiccator, but lose a little during the first two hours at 98°C., thereafter remaining constant, as shown in the earlier paper. The loss at 98°C. was less marked in the fats extracted by petroleum ether than in those by ethyl ether.

Further confirmatory extractions were carried out and the results are summarized in Tables V, VI, and VII, to show any differences in property between the ethyl-ether and the petroleum-ether extracts for the various products examined.

TABLE V

COMPARISON OF FAT EXTRACTED FROM FLOUR BY ETHYL ETHER AND PETROLEUM ETHER

Extract	24 hours in Desiccator		12 hours at 98°C.		24 hours at 98°C.	
	N_D^{20}	Bromine Value	N_D^{20}	Bromine Value	N_D^{20}	Bromine Value
Ethyl Ether	1.4830	67.0	1.4870	....	1.4890	57.0
Petroleum Ether	1.4830	67.5	1.4870	....	1.4890	58.5

TABLE VI

COMPARISON OF FAT EXTRACTED FROM BRAN BY ETHYL ETHER AND PETROLEUM ETHER

Extract.	24 hours in Desiccator		24 hours at 98°C.		36 hours at 98°C.	
	N_D^{20}	Bromine Value	N_D^{20}	Bromine Value	N_D^{20}	Bromine Value
Ether	1.4800	70.0	1.4810	70.0	1.4820	68.0
Petroleum Ether	1.4790	70.0	1.4800	70.0	1.4810	70.0

TABLE VII

COMPARISON OF FAT EXTRACTED FROM GERM BY ETHYL ETHER AND PETROLEUM ETHER

Extract.	24 hours in Desiccator		2 hours at 98°C.		8 hours at 98°C.		20 hours at 98°C.		32 hours at 98°C.	
	N_D^{20}	Bromine Value	N_D^{20}	Bromine Value	N_D^{20}	Bromine Value	N_D^{20}	Bromine Value	N_D^{20}	Bromin Value
Ether	1.4780	82.0	1.4780	....	1.4810	....	1.4840	....	1.4860	65.0
Petroleum Ether	1.4780	81.5	1.4780	....	1.4800	....	1.4837	....	1.4860	61.5

Comparison of Table IV with Tables V and VII shows that a change in weight on heating corresponds with a change in the physical constants of the fat. The weight is constant however after two hours, even with the large amount extracted from the germ, whereas the change in properties continues slowly much beyond that time, the alteration being slower when the quantity of fat is greater. There does not seem, as far as these figures show, to be any appreciable difference in the constants of the products of the two solvents.

Comparison of Various Methods of Extraction

A difference in the amount of fat extracted, depending on the solvent used, has already been seen, and the hydrolysis methods will now be considered. These are the alkaline hydrolysis, the acid hydrolysis, and the alcohol hydrolysis, the last named being the usual method for the estimation of the "lipoids," although the use of this term has been somewhat confused (Maclean, 1926).

Alkaline Hydrolysis Method

The procedure adopted was essentially that given as the tentative method for the estimation of fat in baked cereal products.[1] The only difference between the method employed by the authors and that cited was that the final extract, instead of being decanted, was filtered through a fat-free paper, into the weighed dish.

This modification was made since the authors found that otherwise it was extremely difficult to prevent solid particles being decanted into the weighed dish with the ethereal solution. Provided that the funnel was kept covered during filtration and the paper subsequently well washed with the mixed ethers, no retention of fat by the paper resulted.

In the case of bran, this method gave low results. By using 2 gms. of the sample instead of 5 gms., and by prolonging the time of extraction with the ether portions, however, higher values were obtained.

Acid Hydrolysis Method

In this case the procedure was that of the official method for the determination of fat in flour [2] with slight modifications in respect of two minor points. Firstly, since neither a Rohrig nor a Mojonnier fat extraction apparatus was available, the authors used a stoppered graduated cylinder, the ethereal layer being removed by means of wash-bottle tubes, and secondly, the ethereal extracts were filtered through fat-free paper (with the precautions mentioned under the alkaline hydrolysis method) instead of through cotton.

If a trace of the aqueous solution be carried over with the ether layer, it will appear as a distinct "spot" after the removal of the solvent. Re-extraction with anhydrous ether will dissolve out readily all the fatty material and obviate the error due to the drop of aqueous solution.

In applying this method to germ, good duplicates could not be obtained. After hydrolysis with hydrochloric acid, the germ furnished such a thick, tarry liquid that it was almost impossible to rinse it completely out of the beaker with ether; the separation of the layers in the cylinder was also very unsatisfactory. By using only one gram of the sample, however, these difficulties were lessened and quite good duplicates were obtained.

[1] Association of Official Agricultural Chemists, Methods of Analysis, p. 231, 1925.
[2] Journal of the Association of Official Agricultural Chemists, 9:431-32.

Similar trouble, although not to so great an extent, occurred in the case of bran and again it was found advisable to work with only one gram of sample.

In order to determine whether variation of the time or temperature of the hydrolysis with hydrochloric acid affected the result, the following experiment was performed. A flour was taken and its fat content determined by the acid hydrolysis method but the temperature and time of hydrolysis were varied.

The results are given in Table VIII.

TABLE VIII

EFFECT OF TIME AND TEMPERATURE OF HYDROLYSIS ON FAT EXTRACTED FROM STRAIGHT-RUN FLOUR BY ACID HYDROLYSIS METHOD

Conditions of Hydrolysis (Results Calculated on Dry Basis)	Fat (%)
20 minutes at 70-80°C.	1.98
20 " " 100°C.	2.02
60 " " 70°C.	2.04

Since the figures showed that an increase in the time or temperature of hydrolysis had only a slight effect, the authors decided (as indicated in the details of their method above) to adhere to the time and temperature suggested in the official method, viz., 40 minutes at 70-80°C.

Alcohol Hydrolysis Method

The lipoid content of vegetable material is frequently differentiated from the so-called crude fat content. The former has usually been extracted by an alcoholic hydrolysis method and in this paper the method specified by the Association of Official Agricultural Chemists (Method of Analysis, page 233) as tentative for alimentary pastes has been used.

With bran or germ, however, two grams were substituted for the five grams stated, other quantities being the same—this simplified the manipulation with the bulky material.

Sullivan and Near (1927) have modified the procedure slightly but this modification is primarily for use with dried crude gluten, as low results were obtained on the former method, due to the coherent nature of the material and consequent difficulty of extraction; this binding tendency is not present in the flour, bran, and germ used in the present work. The papers of Sullivan and Near (1927a and 1927b) have shown that there is a greater difference in lipoid content between different grades of flour than between different wheats; the ether extract and lipoid contents of the different flour grades were compared.

The amount of fat obtained from patent and straight-run flour, germ, and bran by the methods under investigation are given in Table IX. These results are all mean values of close duplicates. The figures show that, with the possible exception of bran (see footnote to Table IX), all the samples give higher results by the alkaline hydrolysis method than by direct Soxhlet extraction with ethyl ether; the increase is in the region of 0.5%. The results obtained by the acid hydrolysis method are higher still. In the case of flour, the increase over the alkaline hydrolysis result is not very great but in the case of germ, and possibly of bran also (see footnote again) this increase is considerable.

TABLE IX

FAT CONTENT OF FLOUR, GERM AND BRAN, AS DETERMINED BY VARIOUS METHODS
(Results calculated to ceperntage on dry basis)

Sample	Soxhlet		Hydrolysis		
	Petroleum Ether	Ethyl Ether	Alkaline	Acid	Alcohol ('Lipoid' Content)
Straight run Flour	....	1.53	1.90	1.98	....
Patent Flour	1.16	1.20	1.64	1.70	1.99
Bran	4.18	4.36	4.34[1]	6.73	4.99
Germ	7.82	8.26	8.80	9.62	10.31

[1] This result was obtained by using two grams of the sample and increasing the time of extraction with ether. Using five grams of sample and the normal time of extraction, a mean figure of 4.16 was obtained, i.e. a figure lower than that for the Soxhlet method. Probably by increasing the time of extraction in the alkaline hydrolysis method still more, an even higher result might have been obtained.

Purity of the Fat Extracted by Various Methods

Throughout this paper, the matter extracted by the Soxhlet alkaline hydrolysis, and acid hydrolysis methods has, following the common practice, been referred to as fat by the present writers. It seemed probable, however, that the differences between the results obtained by these three methods might be due, in some part at least, to the extraction in some cases of a certain amount of lipoids. That is, the extracted matter, although referred to as fat, might not always be true fat (i. e. neutral fat and fatty acids) but a mixture of fat and lipoids.

Similarly, the matter extracted in the alcohol hydrolysis method and regarded as lipoids, is in all probability a mixture of lipoids and fat.

In order to test this point, the authors decided to determine the amount of nitrogen and of phosphorus present in the matter extracted from wheaten products by the methods under consideration.

Micro-determination of Nitrogen in So-called Fats

Since the nitrogen contents of these fats were low and the quantity of fat available for analysis often less than 0.1 gm., the actual amount of nitrogen to be estimated was extremely small; this necessitated the use of some form of micro-method.

Preliminary experiments showed, however, that the ordinary micro-Kjeldahl digestions as used for urine, small amounts of purified organic compounds, etc. (Cole, 1926; Pregl, 1924) were useless in the case of fats. On account of their high carbon contents, these substances required to be digested for at least 90 minutes before the solution cleared; even when a few crystals of potassium permanganate were added, the digestion time was not materially diminished. This continued boiling in the presence of such a small quantity of acid furnished low results.

For instance, the nitrogen in about one mgm. of pure dry urea was determined under these conditions, i.e., using 2 cc. of acid and digesting for 90 minutes; 0.000518 gm. of nitrogen was present but only 0.000413 gm. was obtained.

It was thus apparent that as far as the digestion was concerned, this would have to be carried out in the normal manner. On account of the small amount of ammonia that would be present in the digest, it was still essential, of course, that a special method of distillation be employed. The distillation apparatus finally used (see Fig. 1) was a modification of that of Cole (1926). The chief modification was the inclusion of a soda-trap, as the authors found that without this there was a tendency for alkali to be carried over.

The distillation flask is a 300 cc. Kjeldahl flask previously used for the digestion. This is fitted with a rubber stopper carrying a soda-trap and a tap-funnel. The delivery tube of the tap-funnel has a side-tube sealed on just below the tap and this side-tube is connected to the exit tube of the wide-mouthed bottle "D." The inlet tube of this bottle, which reaches nearly to the bottom, is formed into a bulb at the lower end and this bulb is perforated by a number of small holes. The bottle is about two-thirds filled with 10% sulfuric acid in order to absorb any ammonia in the air current.

The soda-trap is connected to a condenser, the other end of which is connected to a tube sealed at the lower end, but with a ring of small holes just above the seal. The stopper through which this delivery tube passes also contains a short tube which is connected to the pump. The receiver "C" is a boiling tube and during

an actual distillation is placed in a beaker of cold water.

With regard to the micro-titration of the distillate, an iodometric method was used. This method was preferred on account of the precautions necessary for the exclusion of carbon dioxide during the titration when standard alkali is employed.

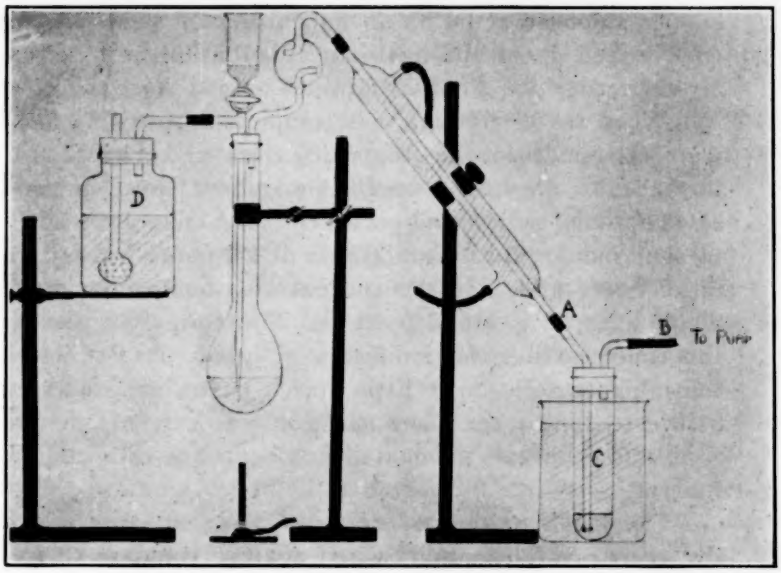


Fig. 1. Apparatus for the distillation of ammonia with aeration.

The actual details of the method as finally adopted are as follows:

> A weighed quantity of the dry fat was washed with ether from the containing beaker into a dry 300 cc. Kjeldahl flask. The ether was then removed by a current of air. Eight gms. of nitrogen-free potassium sulphate, 0.2 gm. copper sulphate, and 15 cc. of concentrated sulphuric acid were added and the flask placed over a low flame. After about 10 to 15 minutes the flame was turned up and digestion allowed to proceed for about 90 minutes. At the end of this time the flame was removed and the flask allowed to cool. The neck of the flask was then washed down with 15 to 20 cc. of ammonia-free water and the flask connected to the distillation apparatus (the apparatus was always thoroughly steamed out with ammonia-free water previous to a distillation). Ten cc. of approximately 0.01N sulphuric acid were run into

the receiver, the stopper and its accompanying tubes inserted and the whole connected to the condenser at "A" and the vacuum pump at "B." A gentle current of air was started through the apparatus and then, by means of the tap-funnel, a 30% solution of caustic soda was slowly added to the contents of the distilling flask until they were alkaline. (The copper sulphate acted as an indicator.) A small flame was then placed under the flask, and distillation in a current of air carried on for 20 to 25 minutes.

When the distillation was completed joints "A" and "B" were disconnected, the stopper of the receiver removed and the tube "C" washed down (inside and out) into the receiver several times with ammonia-free water. Two cc. of 5% potassium iodide solution and 0.5 cc. of 4% potassium iodate solution were added to the contents of the receiver and the whole allowed to stand for 15 to 20 minutes. At the end of this time, the liberated iodine was titrated with 0.01N sodium thiosulphate solution, a little starch paste being added just before the end point. The titration was carried out from a 5 cc. micro-burette graduated in twentieths of a cubic centimeter.

The 0.01N sulphuric acid was made up approximately and then carefully standardized against standard 0.01N sodium thiosulphate. A blank experiment was performed on the reagents used.

This method was applied to the estimation of the nitrogen in about one mgm. of pure dry urea; the actual amount of nitrogen present was 0.000480 gm. and that found was 0.000485 gm.

Determination of Phosphorus in So-called Fats

The small quantity of fat available for a determination combined with the low phosphorus content prevented, in many cases, the tentative Association of Official Agricultural Chemists method for lipoid phosphoric acid from being employed; the weight of magnesium pyrophosphate obtained would have been too small.

Since, however, the amount of the yellow ammonium phosphomolybdate obtained in the first precipitation would be easily weighable, it was decided to employ the principles of von Lorenz' (von Lorenz, 1912; Pregl, 1924) method for the determination of phosphoric acid in fertilizers and soils. In this method the phosphates are weighed as ammonium phosphomolybdate, since the

precipitation is carried out under such conditions that the precipitate is of fixed and definite composition. Since the weight of the yellow precipitate is sixty-eight times that of the phosphorus it contains, the method is particularly accurate. Another important consideration is that a determination can be carried through in two hours. The details of the method as finally adopted for the determination of phosphorus are as follows:

> A weighed quantity of the dry fat was washed from the containing beaker into a platinum capsule with 10 cc. of chloroform in two portions. Ten cc. of 4% alcoholic potash was added to the contents of the capsule and the solution evaporated to dryness on a water bath. The residue was charred over a small flame, care being taken that the contents were not heated above dull redness. When cool, the char was extracted with 5 cc. of dilute nitric acid (1 + 9) and the extract poured on to a small filter. The char was extracted again, this time 2 cc. of the dilute nitric acid being used.
>
> The second extraction was poured onto the same filter. The char was finally extracted with about 5 cc. of boiling water and after this last extract had filtered, the paper was washed once with boiling water. To the combined filtrates, which had a volume of about 15 cc., 2 cc. of the nitric-sulphuric acid mixture (see later) were added and the whole then warmed on a steam-bath. The hot solution was well stirred, 15 cc. of the sulphate-molybdate reagent (see later) poured into the middle of it, the vessel allowed to stand for three minutes, and again stirred for half a minute. The precipitate was then allowed to settle for at least one hour. During this hour, the Gooch crucible was prepared. The layer of asbestos was washed with ammonia, with water, with hot dilute nitric acid, and then again with water.
>
> The water was displaced by washing with 95% alcohol and finally with acetone. The crucible was then wiped on the outside and placed in a vacuum desiccator which contained no drying agent. The desiccator was then evacuated until the pressure was no more than 150 mm. of mercury and maintained thus for half an hour. The crucible was then removed and weighed; the weighing was performed as rapidly as possible. The precipitate, after standing for at least an hour, was filtered off through this crucible. The precipi-

tate was washed twice in the beaker by decantation with 2% ammonium nitrate solution and then washed onto the filter with the same solution.

The beaker was then rinsed out with 2% ammonium nitrate solution and 95% alcohol alternately, until all the precipitate had been transferred to the filter (three washings usually sufficed). A rubber-tipped glass rod was necessary for detaching the precipitate from the sides of the beaker. The filter was then filled twice with 95% alcohol and lastly twice with acetone. After the outside had been wiped, the crucible was replaced in the desiccator, kept evacuated at not more than 150 mm. for half an hour and weighed. The weight of the yellow precipitate multiplied by 0.014524 gave the weight of phosphorus in the material analyzed. (This factor was obtained by Pregl by applying the method to phosphorus pentoxide.)

The reagents used in this method were made up according to the instructions of von Lorenz (von Lorenz, 1912; Pregl, 1924) but the details are given here for reference:

Sulphate-molybdate reagent—

50 gms. of ammonium sulphate was dissolved in 500 cc. of nitric acid of specific gravity 1.36 in a liter flask.

150 gms. of powdered ammonium molybdate was treated with 400 cc. of boiling water in a porcelain dish and stirred until solution was complete. This solution was rinsed into a flask with a little water, cooled to room temperature and poured, in a thin stream with stirring, into the nitric acid solution of ammonium sulphate.

The resultant liquid was diluted to one liter, allowed to stand for two days, filtered, and kept in a well-stoppered bottle of brown glass in a cool, dark place.

Nitric acid containing sulphuric acid—

30 cc. of sulphuric acid of specific gravity 1.84 were poured into one liter of nitric acid of specific gravity 1.19 to 1.21.

Two per cent solution of ammonium nitrate—

This solution was made weakly acid by the addition of a few drops of nitric acid per liter.

Table X gives the percentage of nitrogen contained in the fatty bodies (after drying) extracted from various wheaten products by different methods.

TABLE X

PERCENTAGE OF NITROGEN IN FATTY BODIES EXTRACTED FROM WHEATEN PRODUCTS BY DIFFERENT METHODS

(Results obtained on dry material)

	Soxhlet		Hydrolysis		
Sample	Petroleum Ether	Ethyl Ether	Alkaline	Acid	Alcohol
Patent Flour	0.88	0.68	0.78	0.20	1.15
Bran	....	0.11	0.45	0.19	0.52
Germ	....	0.08	0.16	0.16	0.43

Table XI gives the phosphorus contents of the same dried fatty bodies.

TABLE XI

PERCENTAGE OF PHOSPHORUS IN FATTY BODIES EXTRACTED FROM WHEATEN PRODUCTS BY DIFFERENT METHODS

(Results obtained on dry material.)

	Soxhlet		Hydrolysis		
Sample	Petroleum Ether	Ethyl Ether	Alkaline	Acid	Alcohol
Patent Flour	0.30	0.27	1.14	0.21	1.07
Bran	....	0.11	0.48	0.13	0.34
Germ	....	0.28	0.53	0.15	0.48

These figures show that the fatty bodies extracted by the acid hydrolysis method contain less nitrogen and phosphorus than those extracted by the other hydrolysis methods. This presumably indicates that this method gives the purest fat of any considered in this paper—in other cases the extracted fatty substance is more or less contaminated with nitrogen and phosphorus-containing material. From this aspect, therefore, it seems desirable to conform with the Association of Official Agricultural Chemists decision to apply this acid hydrolysis method to flour and its chief baked product—bread.

Comparison of Amounts of Fat Extracted From Flour and the Bread by the Acid Hydrolysis Method

The method as given above was applied to a series of flours and to the breads made from them. The flours were of different commercial grades from a South of England mill and were all from the same grist. The bread was made on a laboratory test scale by a skilled test baker on the procedure given by Kent-Jones (1927,

p. 174), in which no shortening was used. Cottage loaves were made of the white flours but a tin loaf from the wholemeal.

Moistures were determined at the same time and the results, given in Table XII, are calculated on the dry basis.

TABLE XII.

COMPARISON OF QUANTITY OF FATTY BODIES EXTRACTED FROM FLOUR AND THE CORRESPONDING BREAD BY THE ACID HYDROLYSIS METHOD

Calculated to dry basis.

Grade	Amount of Fat Extracted	
	Flour %	Bread %
Patent (Top 50%)	1.90	1.92
Straight Run	2.29	2.19
Low Grade (Bottom 50%)	2.48	2.54
Wholemeal	2.81	2.92

The agreement is seen to be fairly satisfactory.

Discussion

The suggestion that the supposed change in the fat extracted from cereal products on heating was due to the removal of traces of water and solvent, has been examined. There appears to be a definite point reached in a vacuum, both as regards weight of fat and its properties; on drying at the temperature of boiling water changes occur in the physical constants and further weight is lost. This would conform to the suggested effect of removal of traces of solvent, but a comparison of Tables IV, V, and VII shows that, whereas the weight remains constant after two hours' heating at 98°C., the changes in the physical properties continue, and that the rate of change depends upon the total quantity of fat present. Further, the loss in weight on drying for two hours at 98°C. is less with the petroleum-ether extract than with the ethyl-ether extract, but the changes in physical constants are quite parallel. These figures appear to suggest that, although minute traces of water are retained in the fat before drying at a higher temperature, these are not sufficient to account for the changes noted in the constants.

It has been quite definitely shown that the amount of fatty matter estimated depends to a very large measure on the method used for its extraction. Even the substitution of one solvent for another makes an appreciable difference in the result; it is imperative, therefore, that some knowledge should be obtained as to which method gives the best indication of the amount of pure fat obtainable.

It is customary both in England and in America to differentiate the so-called fat or crude ether extract from the lipoid or alcohol-ether extract, and the introduction of the hydrolysis methods of estimating fat has confused the matter. It can be presumed that the ether extract contains more or less the free fat (neutral fat and fatty acids) existing in the product under examination, whereas the hydrolysis methods will extract, as well as this free fat, some of the combined fats, which have been liberated by the hydrolysis; the extent to which these fat-containing compounds are split will depend on the severity of the hydrolysis and may vary in the different methods. The classification of the extract by the alcohol hydrolysis method as lipoids, suggests that, by this method, only the fat in combination is obtained, but the authors fail to see why this is not a mixture of the free fatty bodies together with those liberated from lipoid combination.

Sullivan and Near (1928) state that this alcoholic-ether extract includes phosphatides, neutral fats and small amounts of fatty acids, chlorophyll, and sterols. Similarly, the other hydrolysis methods will presumably estimate together these same fractions.

TABLE XIII

GRAMS OF NITROGEN AND PHOSPHORUS EXTRACTED FROM 100 GRAMS SAMPLE (DRY BASIS) BY VARIOUS METHODS

Sample	Soxhlet		Hydrolysis		
	Petroleum Ether	Ethyl Ether	Alkaline	Acid	Alcohol
lour Phosphorus	0.0035	0.0032	0.0187	0.0036	0.0213
" Nitrogen	0.0102	0.0082	0.0128	0.0034	0.0229
Bran Phosphorus	.....	0.0048	0.0208	0.0087	0.0170
" Nitrogen	.....	0.0048	0.0195	0.0128	0.0259
Germ Phosphorus	.....	0.0231	0.0466	0.0144	0.0495
" Nitrogen	.....	0.0066	0.0141	0.0154	0.0443

The examination of the material obtained by all these methods for nitrogen and phosphorus was therefore undertaken and the results have been given in Tables X and XI. Owing to the different amounts of extract obtained, for ease of comparison the quantities of nitrogen and phosphorus in them have been recalculated to the amount extracted from 100 gms. of the dry wheaten product. These figures are given in Table XIII. Determinations of the phosphorus content of the ether extracts and of the ether-alcohol (lipoid) extracts of different mill stocks were made by Sullivan and Near (1928), who showed that the higher grades of flour contain more phosphorus in the lipoid extract than do the lower grades and

offals; also that the ether extract in all cases shows a lower phosphorus content than does the lipoid extract. The nitrogen percentage in the lipoids is shown by them to be in the same order as the phosphorus content, that is, the patent flour, although having the least total nitrogen, contains more of this element in its lipoids than do the lower grades.

From this table the following information can be deduced:

(1) The Soxhlet ether extraction gives a fat of fair purity as regards nitrogen and phosphorus, save in the case of germ, when the phosphorus is high.
(2) Of the hydrolysis methods, the acid hydrolysis gives the fat containing least nitrogen and phosphorus; in the case of germ, however, the nitrogen content with the alkaline method is slightly lower than that with the acid.
(3) The alkaline and alcohol hydrolysis give fairly similar results.
(4) The ether extraction and acid hydrolysis methods also give fairly similar results in respect to nitrogen and phosphorus.

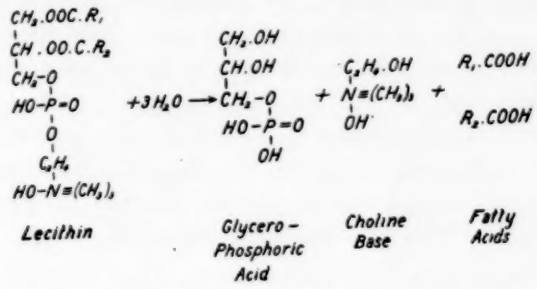
It was provisionally suggested in an earlier part of the paper that the quantity of fat extracted might be dependent upon the degree of hydrolysis, and the results obtained show

(a) that larger quantities are extracted by *all* the hydrolysis methods, the acid being slightly higher than the alkaline.
(b) that the acid method gives the purest fat.

In order to interpret these results, it is necessary to consider the constitution of these nitrogen- and phosphorus-containing bodies, namely, the lipoids. The graphic formula of a typical lipoid, lecithin, appears below where R_1 and R_2 stand for similar or dissimilar fatty acid radicals. Various workers, using lecithin from different sources, have obtained different fatty acids upon hydrolysis, e.g.: oleic, palmetic and stearic acids have been isolated, and, in the case of Soya bean lecithin, linolic, and linolenic acids were obtained in addition (Maclean, 1926).

These lipoids are normally found in plant products in combination with protein materials (see Sullivan and Near, 1928), although Taylor and Nelson (1920) have shown that a combination exists in maize, at any rate, between a carbohydrate and a fat, but owing to the lack of information available on this, it has not been included in the following discussion. It is agreed that on hydro-

lysis the compound molecule will break down, firstly into lecithin and protein and subsequently into the choline base, glycero-phosphoric acid and the fatty acids (Maclean, 1926).



It may, therefore, be suggested that the alkaline hydrolysis and the alcohol hydrolysis effect the first stage, but that the acid method produces, at any rate partially, the second stage. Of course, it is probable that with any method there is no hard and fast line of separation between the two hydrolytic processes.

If this assumption be correct, then an explanation is given for the apparent anomaly that the extract from the acid hydrolysis contained less nitrogen and phosphorus than those from the other hydrolysis methods.

It was thought to be of interest to calculate approximately the phosphorus content to lipoids, taking the above lecithin formula as representative, and assuming the fatty acid radicals (R_1 and R_2) to have a molecular weight of 238. Stearic acid radical has a molecular weight of 239 and oleic acid radical 237; further, Bailey (1925) gives the molecular weight of the fatty acids in bran as 285, which would correspond to a radical of relative weight 240.

On this assumption the molecular weight of the lipoid will be $329 + (2 \times 238) = 805$. After converting the phosphorus content of the extracts to the equivalent weight of lipoids, the residual nitrogen, after subtracting that required for the calculated lipoids, was calculated to protein (using the nitrogen-protein conversion factor of 5.7). The calculated lipoid and protein contents subtracted from the original extract obtained gave a figure which was called by the authors "free fat." It is realized, of course, that these calculations and assumptions are purely arbitrary, but a study of Table XIV, in which they are reported, is of interest.

On these assumptions the amount of protein extracted is low throughout. The alkaline and alcoholic hydrolysis methods apparently extract largely the lipoid compound as such, while this is

not the case in the acid hydrolysis method. Some evidence is therefore deduced that the acid hydrolysis goes in the main a stage further than the other hydrolysis methods and actually may break down the lipoid molecule into its hydrolytic components. In the case of flour, it might be deduced from Table XIV that the amount of free fat pre-existing in the sample is approximately 1.10% and the total of the original free fat and the lipoid fat is 1.60%; if the difference 0.5% is calculated to lipoid, this would correspond to 0.65% lipoids, indicating that the major portion of the lipoid material is obtained unhydrolyzed in the alkaline and alcoholic hydrolysis methods. This procedure of deduction is not so simple in

TABLE XIV

ASSUMED DISTRIBUTION OF NITROGEN AND PHOSPHORUS IN FATTY EXTRACTS FROM 100 GRAMS DRY CEREAL PRODUCTS

	Soxhlet	Hydrolysis		
	Ethyl Ether	Alkaline	Acid	Alcohol
		Flour		
Lipoids	0.08	0.49	0.09	0.55
Protein	0.04	0.03	0.01	0.08
Free Fat	1.08	1.12	1.60	1.36
Totals	1.20	1.64	1.70	1.99
		Bran		
Lipoids	0.13	0.54	0.23	0.44
Protein	0.02	0.06	0.05	0.10
Free Fat	4.21	3.74	6.45	4.45
Totals	4.36	4.34	6.73	4.99
		Germ		
Lipoids	....	....	0.37	1.29
Protein	....	....	0.05	0.12
Free Fat	....	....	9.20	8.90
Totals	....	....	9.62	10.31

the cases of bran and germ, particularly in the latter case, where the nitrogen content of the extract is not sufficient to fulfill the lipoid requirement of the phosphorus (see Table XIII).

Recapitulating, the authors suggest that the difference between the estimation of fats and lipoids is generally too much emphasized and that the various methods in the literature give the sum of the free fat plus a larger or smaller proportion of the lipoid material, which may or may not be hydrolyzed.

It is probable that the Soxhlet extraction does give the nearest approximation to a measure of the free fat existing in the product, and that the normal method for lipoids includes fat and unhydrolyzed lipoids, but the acid hydrolysis gives the total fat of the product; i. e. the pre-existing free fat plus the fat liberated from its combination.

Summary

Ether-extracted fats lose in weight on heating at 98°C. for two hours, but thereafter remain constant.

The physical constants of these fats change progressively on heating at 98°C. after the loss in weight has ceased.

The various methods for the estimation of fatty materials and lipoids are compared, and it is shown that the hydrolysis methods invariably give higher results than direct extraction methods.

The direct extraction and the acid hydrolysis methods give products more free from nitrogen and phosphorus than do the other methods. It is suggested that the acid hydrolysis method may hydrolyze, partially at any rate, the lipoid molecule.

The acid hydrolysis method gives comparable results on flour and the bread made from it.

It is finally suggested that the direct ether extraction will give a measure of the free fat existing in the product, but that the acid hydrolysis method gives the total fat content, that is the free fat plus the combined fat, after liberation from its combination.

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A COMPARATIVE STUDY OF THE NON-GLUTEN CONSTITUENTS OF SOFT AND HARD WHEAT FLOURS[1]

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(Received for publication, October 30, 1929.)

It has been the custom to evaluate all flours by means of routine tests which have for their objects the determination of such constituents as ash, moisture, protein, wet and dry gluten, etc. How far these determinations do define baking quality in a cake flour has yet to be proved. Indeed there appears to be no universal "yardstick" on which to measure this property.

In bread baking, the gluten undoubtedly is the "framework" of the loaf, but does this apply in like degree in cake making? In order to make a comparison let us examine a commercial bread formula side by side with commercial cake formulae. Bread flours have usually about 11% dry gluten and cake flours around 8% dry gluten. These figures will suit our purpose for this comparison, and in adopting them 100 lbs. of bread flour will therefore add 11 lbs. of dry gluten to the dough batch, while 100 lbs. of cake flour will add only 8 lbs. of dry gluten.

	Batch No. 1 Bread	Batch No. 2 Pound Cake	Batch No. 3 Layer Cake	Batch No. 4 Sponge Cake
Flour	100	25	33	26
Dry gluten	11	2	2.6	2.0
Water	60	..	..	..
Yeast	2	..	..	..
Salt	2	..	..	..
Sugar	2	25	22	34
Malt	1	..	..	..
Shortening	2	25	11	..
Milk Powder	2	..	..	..
Eggs	..	25	20	40
Milk	..	..	14	..
Total Bulk	171	100	100	100
Dry Gluten	11 = 6.4%	2%	2.6%	2.0%

[1] This work was carried out at Trent Institute of Baking Technology, Ontario Agricultural College, Guelph, Ontario, Canada, under the H. E. Trent and Fleischmann Scholarships.

Bread batch No. 1 has a bulk of 171 lbs. and contains 11 lbs. dry gluten which expressed in percentage = 6.4% dry gluten; No. 2, Pound Cake, has a bulk of 100 lbs. and contains 2.0% dry gluten; No. 3, Layer Cake, has a bulk of 100 lbs. and contains 2.6% dry gluten; No. 4, Sponge Cake, has a bulk of 100 lbs. and contains 2.0% dry gluten.

It will readily be perceived that a bread batch contains nearly three times the amount of dry gluten that a normal cake batch contains.

In view of this fact, can we maintain that the routine tests which define baking quality in the former also hold good for the latter?

Reviewing our figures for cake batches Nos. 2, 3, and 4, it is at once apparent that by deducting the percentage of dry gluten from the total flour content, we will determine the amount of what we could properly describe as the non-gluten portion of the flour ingredient.

Expressed in percentage ratio of the total batch, they are:

	Total Batch	Non-gluten portion of flour
No. 2, Pound Cake	100	23%
No. 3, Layer Cake	100	30%
No. 4, Sponge Cake	100	23%

The non-gluten constituents of a normal cake batch, then, will represent roughly, about one-fourth of the total batch.

Can we continue to regard this non-gluten portion of the flour ingredient as something inert, something that merely acts as a filler?

A study of the non-gluten properties of soft wheat flour should help to elucidate this point.

In making an examination of hard spring wheat flour and soft winter wheat flour under the microscope, one is particularly impressed with the striking differences in the general appearance of each. The former presents a clean-cut appearance with the starch granules embedded in the gluten matrix, while the latter appears to be composed chiefly of aggregates of starch granules, held together much more loosely by a significantly smaller gluten matrix, and also, in the latter, there is an abundance of free starch granules, which is a strong point of difference in comparison with flours from hard spring wheats. Indeed by adjusting the angle of the mirror to secure a dark background, the dry flour particles are seen as shining white particles, which, in the case of the hard spring wheats may aptly be described as presenting the appearance of particles of ice, while those of the soft winter wheats have a soft woolly appearance, which to carry out the previous simile, may be said to strongly resemble snowflakes in appearance.

Patterson (1924) in treating this subject of cake flours, states that while the relation of gluten quality and quantity are important considerations, an "ideal" cake flour seems to be one in which *each individual starch granule is separated from its neighbour,* and the writer has found from microscopic examinations that the nearer we approach to this "ideal" the better are the results we obtain in cake shop practice, all of which suggests the importance of free starch in cake flours.

In this regard, we are again reminded of a further difference between hard spring, and soft winter wheat flours, in the treatment they receive in their respective processes of manufacture into baked products. In the manufacture of bread doughs, we usually allow an addition of 60% added moisture, based on 100% flour, while in cake-making the moisture content of the batch for layer cakes is usually around 90% to 100% and that for jelly rolls, etc., over 100%. If a dough of flour and water be made using 100% of each we produce something which could more correctly be called a batter, from which we cannot obtain gluten by washing. Swanson and Working (1926, p. 78), find that in the presence of excess water no gluten formation is apparent, but gluten can be obtained only if the excess water is removed by centrifugal force.

If this be true, then, a normal cake batter is one which does not lend itself readily to the formation of gluten, and in view of the fact that soft wheat flours contain only about 7% to 9% gluten we are reminded of the possibility that the non-gluten constituents, both on account of their bulk and the other considerations noted, may play a part in determining that something we speak of as "quality" in a cake flour.

The very fact that corn starch may replace cake flour entirely in a loaf cake type batch and still produce that characteristic cake-like "mealy" texture in the finished product, helps to strengthen our theory that the non-gluten constituents of cake flours are important.[1]

Alsberg (1928) says that "though starch is nearly four-fifths by weight of flour, the literature upon wheat, milling flour, and baking, pays but scant attention to the role of starch in dough and bread." The same writer dealing with granule size and heat gelatinization points out that wheat starches do not burst and disinte-

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SAND CAKES

[1] 1 lb. powdered sugar; 14 only eggs; 15 ozs. corn starch; 4 ozs. melted butter. Beat eggs and sugar 5 minutes at 110° F. fast speed. Fold in the starch carefully and when almost clear add the melted butter.

grate when boiled, but the granules merely swell without losing their individuality. Upon this he draws the inference that the physical properties of *gelatinized starch depend upon the size to which they swell,* and the volume they can occupy as compared with the total volume of the liquid in which they are suspended.

Katz (1928) working on changes in bread upon staling, points out that there are two grades or orders of gelatinization of starches.

In the first order, starch with 50% water added and gelatinized at 100°C. for one hour, remains as a white powder *but each individual starch granule has increased in size.*

In the second order of gelatinization however; that is, in the presence of an abundance (100% and over) of moisture at 100°C. for one hour, the whole mass becomes transparent and opalescent, and the granules are intensely swollen.

Undoubtedly, gelatinization of the first order takes place when bread is baked, but on account of the increased moisture content of a cake batch, one would look for a type of gelatinization merging upon the second order when a cake is baked.

In order to determine this point, 1 gm. of air dried wheat starch (from soft flours) was mixed with 1.0 cc water and gelatinized for one hour at 100°C., and 1.0 gm. mixed with 0.5 cc. water and gelatinized in the same manner, the former reproducing very nearly the conditions existing during the baking of a cake batter, and the latter of bread dough.

When examined after gelatinization it was noted that in the case of the one with 50% water the product was quite white in colour, while in the case of the one with 100% water, the white colour had disappeared and the product was more translucent.

A microscopical examination showed the granules in the 100% moisture test to be almost twice as swollen as those in the 50% moisture test.

Starches from the crumb of a loaf of bread and the crumb of a cake, examined before and after staining with Lugol's solution (I-KI) show a similar difference, the largest granules in the former measuring 35 to 45μ and in the latter " to 65μ.

From the foregoing, the fact that starch is more completely gelatinized in cake than in bread would seem to be established.

In working with different starches in the second order of gelatinization, the writer noted that some starches swell to higher volumes than others.

Rask and Alsberg (1924) suggest, in a viscometric study of starches, that there may be a possibility of developing a method for distinguishing winter and spring wheat starches, as winter wheat starches have higher viscosities than spring wheats.

The writer has found that those starches which have high viscosities also have high swelling powers in the second order of gelatinization.

It was determined, therefore, to examine this phenomenon of swelling power of starches, reproducing as nearly as possible the conditions of moisture and temperature which exist in a cake batter when being baked.

In the first place it was necessary to determine the exact maximum temperature which obtained in the centre of a cake during baking. This was recorded on maximum thermometers, specially made by our Physics Department, of such dimensions as to be completely enclosed within the centre of the cake during baking, and averaged 99°C.

It was also proposed to keep the moisture content of each test to the same proportion as the normal moisture content of a cake batter. Several difficulties appeared in this regard, however. One gram of moisture-free air-dried starch was gelatinized with 1 cc. H_2O at 100°C. for one hour in a 10 cc. graduate. It was very difficult to secure uniform results as some of the starch adhered to the cylinder as dust, and again some of the starch suspension adhered to the sides after mixing with a needle, and for these reasons absolutely concordant results were not obtained.

It must also be noted here that Reichert (1913), LeWall and Graves (1913) and Nyman (1912) find that starch which has been dried has a different gelatinizing temperature from that freshly isolated from the living tissue.

In order to obviate these objections it was decided to wash out the starch in the ordinary manner and to secure uniformity the following modifications were adopted:

The room, flour, and distilled water were kept at a constant temperature of 25°C.

Twenty-five grams of moisture-free flour was made into a dough with distilled water taken from a 500 gm. portion previously weighed in a tared vessel and transferred to a 500 cc. burette, and *this amount only was used for washing and doughing.*

The burette was clamped at a convenient height above a crock and the flow of water adjusted requisite to the requirements of the washing process.

The dough was completely enclosed in a previously tared 9-inch square of washed and dried cheesecloth, and the washing carried to completion with the balance of the water. Cheesecloth was used because of the difficulty experienced in collecting the gluten from soft flours when using distilled water.

The completeness of washing was checked by transferring portions of the starch suspension from the crock to a tared beaker from time to time, so that in the final stages the water became less and less milky until finally it remained clear.

The wet gluten was freed of as much moisture as possible by squeezing in the hand and then dried to constant weight at 100°C., and weight of dry gluten determined by deducting tare weight of cheesecloth.

The whole of the collected starch suspension was then weighed, and, by careful manipulation throughout many determinations, approximated 506 gm. so very closely that this was regarded as the average weight of the starch suspension for the washing process. Any great variations were regarded as unreliable but by careful attention to details very uniform results were recorded.

These suspensions were then filtered on tared filter papers which had been dried to constant weight. The residue was then air dried and afterwards dried to constant weight in a moisture oven at 102°C., which usually required 14 to 18 hours. The weight of the dry residue was then recorded which throughout a large number of duplications proved to be very constant. Weighing of this residue, however, had to be done with reasonable speed, as the starch thus treated began to increase in weight when exposed to the atmosphere.

After it was found that concordant results could be obtained with care, both in the weight of total suspension and in the resultant residues, duplicate washings were made and suspensions checked by weighing as noted.

The suspension was then uniformly dispersed by pouring from one beaker to another alternately and while it was thus agitated 10 cc. aliquots were taken up by the means of a pipette and immediately transferred to 10 cc. graduated cylinders until 18 cylinders had been so treated. Two of these were gelatinized immediately, while uniformly dispersed by placing the cylinders upon an asbestos pad in a constant temperature oven at 100°C. for one hour.

Others were allowed to settle for varying periods up to 16 hrs., and then gelatinized in duplicate under the same temperature and time conditions (See Table I).

Those which were allowed to gelatinize while suspension was completely dispersed gave consistently higher volumes of the gelatinized suspension residues than those allowed to stand for varying periods, thus enabling the suspensions to settle at the bottoms of the cylinders.

In order to ascertain if results were influenced by variations in quantities of suspension, two 100 cc. graduates were filled in a similar manner and the same procedure for gelatinization followed.

TABLE I

VARIATIONS IN VOLUME, WEIGHT, AND CONSISTENCY OF THE NON-GLUTEN CONSTITUENTS OF SOFT WINTER WHEAT FLOUR NO. 5 UNDER VARYING PRE-GELATINIZATION.

Suspension Gelatinized 1 hour 100° C.	Pre-gelatinization Resting Period	Volume cc.	Weight grams	Surface Condition
	Resting Periods			
10cc	none	2.80	2.80	watery
"	none	2.80	2.80	watery
"	15 minutes	2.45	2.48	watery
"	15 minutes	2.45	2.47	watery
"	30 minutes	2.20	2.21	viscous
"	30 minutes	2.20	2.20	viscous
"	45 minutes	2.15	2.14	plastic
"	45 minutes	2.15	2.14	plastic
"	60 minutes	2.00	2.05	firm
"	60 minutes	2.00	2.05	firm
"	120 minutes	2.00	2.00	firm
"	120 minutes	2.00	2.00	firm
"	180 minutes	2.00	2.00	firm
"	180 minutes	2.00	2.00	firm
"	720 minutes	1.90	1.90	firm and solid
"	720 minutes	1.90	1.90	firm and solid
"	960 minutes	1.90	1.90	firm and solid
"	960 minutes	1.90	1.90	firm and solid
100cc	720 minutes	19.00	19.00	firm and solid
"	720 minutes	19.00	19.00	firm and solid

The results obtained were concordant with those when smaller quantities were used. It was noted that those suspensions which had not been allowed to settle previous to gelatinizing had a soft watery surface and when the supernatant liquid was poured off part of this surface poured off with it and was noted to be quite viscous and semi-fluid.

After standing one hour and then gelatinizing it was observed that the surface area of the gelatinized portion became more definite and solid and at 12 hrs. standing before treatment in the oven, the residues were uniformly definite and firm and the supernatant liquid poured off without disturbing the gelatinized residue.

After the graduates had been taken from the oven and allowed to cool at room temperature, the surface liquid was poured off and

volume of solid residue recorded. The weight of this was then determined and in every case volume and weight were synonymous.

For example, if a suspension had swelled to a volume of 3.5 cc. the weight would be 3.5 gms. approximately. Likewise if a suspension had swelled only to 1.5 cc. as after 12 hrs. standing, the weight also would approximate 1.5 gms.

A duplicate washing was made from which no gelatinization tests were made, but the whole was filtered, air-dried, and dried to constant weight.

This weight of dry residue in the total volume of suspension was used in computing the amount contained in each of the 10 cc. graduates by weighing them, and the difference in weight between this computed weight and the weight of the residue gelatinized was then regarded as weight of water imbibed during gelatinization. If 506 gms. total suspension contains 19.6 gms. dry starch residue, 10 gms. will contain $\frac{19.6 \times 10}{506.0}$

The volume to which a suspension swells may therefore be regarded as an index of its swelling power and the increase in its weight may likewise be regarded as its moisture-imbibing power.

Comparisons were made in the manner previously described between soft wheat flours and hard wheat flours (See Table II).

Twenty-six flours were examined and results tabulated, 8 of which were commercial samples of flours sold for breadmaking in Canada and 16 were commercial cake flours drawn from the United States and Canada, while two were milled on an Allis Chalmers experimental reduction machine from two soft wheats grown on the experimental plots of the Ontario Agricultural College.

Each experiment conducted was repeated several times in order to establish its accuracy and to check any errors due to technique.

All gelatinization tests were given a 12 hr. pre-gelatinization resting period, because at this period, results were found to be unvaryingly uniform.

Flour was the only variable, all other factors being kept constant so that differences in results should represent actual differences in the non-gluten constituents of the various flours examined.

The differences noted are of such a nature as to warrant further work, therefore a series of baking tests are in progress, results of which will be made subject matter for a future paper.

Two 10 cc. aliquots were rested two hours, then gelatinized for two hours under the same conditions of temperature, when a

series of pockets (presumably steam) formed at the base of the cylinders which forced the solid gelatinized residue up the graduate slightly. This, however, receded on cooling and the volume noted was again concordant with previous resting and ovening period results.

TABLE II

COMPARISON BETWEEN HARD AND SOFT FLOURS — SHOWING DIFFERENCES IN SWELLING POWER AND WATER-IMBIBING POWER OF THE NON-GLUTEN CONSTITUENTS, AND CHEMICAL ANALYSIS.

	500 gms. H_2O, 25 gms. moisture free flour								
Flour No.	Total Suspension, grams	Dry Gluten, grams	Dry Residue grams	Loss[1] Dry Solids, grams	10 cc. Graduates Gelatinized Volume, cc.	Weight, grams	Ash (13.5% Moisture Basis) %	Protein %	Moisture %
1 S	506.5	3.05	19.75	2.2	1.50	1.50	.42	9.50	10.0
2 S	506.0	2.50	20.30	2.2	1.60	1.58	.40	8.80	10.1
3 S	506.0	2.75	20.10	2.15	1.55	1.57	.40	8.80	11.1
4 S	507.0	2.48	20.41	2.11	1.70	1.71	.43	9.00	9.5
5 S	505.5	2.00	20.40	2.60	1.75	1.75	.39	7.40	9.3
6 S	506.5	2.63	20.19	2.18	1.50	1.51	.38	8.70	9.6
7 S	506.5	2.63	20.05	2.30	1.55	1.55	.40	9.00	9.4
8 S	505.0	2.65	20.20	2.15	1.55	1.56	.44	9.50	9.6
9 S	506.0	1.95	21.05	2.00	1.85	1.85	.37	7.80	8.8
10 S	507.0	2.45	20.40	2.15	1.85	1.90	.435	9.00	10.8
11 S	506.0	2.40	20.45	2.15	2.00	2.05	.44	9.10	10.8
12 S	506.0	2.18	20.60	2.22	1.90	1.90	.39	8.20	9.3
13 S	505.0	2.90	19.80	2.30	1.50	1.50	.43	8.90	10.3
14 S[2]	506.0	2.25	19.79	2.96	1.50	1.50	.41	9.05	10.9
15 S[2]	507.0	2.26	19.75	2.91	1.50	1.50	.41	9.00	10.0
16 S	506.0	2.75	20.00	2.25	1.55	1.56	.44	9.10	12.0
17 S	507.5	1.90	21.29	1.90	2.00	2.00	.35	7.00	11.8
18 S	506.0	1.96	20.99	2.05	1.85	1.80	.35	7.40	11.8
19 H	505.8	3.00	19.60	2.35	1.40	1.40	.39	11.85	12.6
20 H	506.5	3.05	19.50	2.40	1.30	1.29	.39	11.75	12.7
21 H	506.2	3.10	19.45	2.40	1.40	1.41	.47	11.85	12.3
22 H	505.6	2.99	19.50	2.51	1.25	1.25	.51	11.59	11.2
23 H	506.0	3.05	19.55	2.40	1.35	1.34	.45	11.80	12.1
24 H	506.5	3.20	19.38	2.42	1.30	1.31	.50	11.60	12.5
25 H	505.5	3.09	19.50	2.41	1.40	1.38	.46	11.90	11.8
26 H	505.5	3.15	19.40	2.45	1.30	1.28	.50	12.00	11.8

[1] Loss in manipulation obtained by subtracting weight of dry gluten plus dry residue from weight of original moisture-free-flour.
[2] Contained potato starch.

General Summary of Conclusions

A normal cake batter contains approximately only one-third the amount of dry gluten as compared with bread.

In washing the gluten of a soft flour it is impossible to collect the gluten if sufficient water is added to the dough to approximate the moisture content of a cake batter.

A normal cake batter does not readily lend itself to gluten formation, as in the presence of excess moisture this formation is not apparent.

It is possible to produce "mealy" cake-like texture in a certain type of cake which is made entirely from cornstarch in which no gluten is present.

The non-gluten portion of the flour ingredient in cake represents one-fourth of the total bulk.

Starches in cakes are more completely gelatinized than starches in bread.

High viscosities of the gelatinized non-gluten constituents appear to be associated with high swelling powers and high water-imbibing powers.

Maximum baking temperatures recorded within the centre of cakes of various types were: Layer cakes, 98°C.; Loaf cakes, 99°C.; Sponge cakes, 99°C.

A pre-gelatinization rest of 12 hours gave consistent results throughout.

Variations in quantities of suspension used did not influence results when given a pre-gelatinization rest of 12 hours.

The non-gluten constituents of soft wheat flours are present in higher ratio than those of hard wheat flours.

The non-gluten constituents of soft wheat flours have proportionately higher swelling powers and moisture-imbibing powers than those from hard wheats.

The differences noted in swelling power and water-imbibing power of non-gluten constituents from different flours gelatinized under identical conditions are of such a nature as to warrant further work, therefore a series of baking tests are in progress, results of which will be made the subject matter for a future paper.

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CONCERNING POSSIBILITIES OF STANDARDIZING THE GRANULATION TEST FOR FLOUR[1]

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(Received for publication October 21, 1929)

To have an understanding concerning the granulation of flour is knowledge which the baker may apply to good advantage in his business, as the degree of fineness and uniformity of flour particles has, normally and under exaggerated conditions, a marked effect on fermentation and on the quality of the finished product.

Bakeshop conditions may be such that it is difficult to regulate temperature, particularly above normal, and knowledge concerning the granulation of flours and their behavior will prove helpful in meeting difficulties which may be encountered.

As progress has been made in the industry the question of economizing on time and labor has required much consideration. One outcome in the baking industry has been the development of the no-time dough. To allow for the short fermentation and at the same time to obtain the best possible results it will prove an advantage to know which kind of granulation (degree of fineness) will be most adapted to the purpose.

Naturally it is up to the miller to produce flour to meet the demands of his trade, which is subject to changes from time to time.

Previous investigation has dealt with the influence of the size of the flour particles on the yield, composition, uniformity and characteristics of products as well as on the baking quality of flour.

Referring to literature published in America: No reference is made by Le Clerc and his coworkers (1919); Shollenberger, Marshall and Hayes (1921); Dedrick (1924); Shollenberger, Marshall and Coleman (1924); Alsberg and Griffing (1925); Kress (1928),

[1] Published as Technical Paper from Trent Institute of Baking Technology with the approval of the President of Ontario Agricultural College, Guelph, Canada.

which would suggest that these authors met with any difficulties as regards separation of the particles. This is not the case with Shollenberger and Coleman (1926) who found that "Although the portions ground the most number of times were undoubtedly the finest, yet in many instances a smaller proportion of the material ground twenty times sifted through the 25XX silk cloth than of material which was ground ten times, four times, or even once. This result was probably due to the fact that the materials ground twenty times became feathery and greasy in character, which made them the more difficult to sift."

In Germany research has been carried on which has dealt directly and indirectly with granulation. Attempts have been made to standardize a method of control in the flour mill by means of stock analysis, that is, the sifting out of intermediate products on a set of sieves. [Scherz (1927), Haltmeier (1927, 1928), Fornet (1928).] The application of a similar control of stock analysis has for decades served as a check on breweries, and other pulverizing industries are employing this method in an endeavor to control the quality of their product. [Rammler (1927), Perrott and Kimney (1923).] In using stock analysis as a means of control in the flour mill, difficulties presented themselves as to the individual interpretation and application of results. Haltmeier (1928) after making microscopic studies of the different bolting cloths, (X, XX, XXX) found that with precise knowledge concerning the net apertures of the meshes it was possible not only to interpret the results of stock analysis but that such results were applicable as a means of control in flour mills, and moreover would entirely eliminate the personal factor of the operator. The proposed graphic method of arranging the results of stock analysis makes them independent of the set of sieves used, and consequently readily comparable.

Foerderreuther (1928) claimed that silks, due to discrepancies with extremely fine silks, were less dependable for stock analysis than wire cloths.

Kettenbach (1928) came to the conclusion "that stock analysis furnishes a lucid survey of the breaking-up process and enables us to choose the proper sifter cloths, in a manner and to a degree unsurpassed by any other known method."

Pfister (1928) maintained that granulation should be determined by the help of a small motor-driven laboratory sifter and that the minimum time of sifting should be 30 minutes. Furthermore, Pfister claimed that all the flour would pass through the

finest silk if the sifting process was continued long enough. He also emphasized the need of standardization of the granulation test.

Van der Lee (Dutch investigator, 1928) experienced great difficulty in separation of flour particles and expressed the opinion that this was chiefly due to certain electric potentialities which were made manifest through friction, as a result of which he considered it practically impossible to thoroughly separate the fine particles.

In America the granulation test is often asked for, some millers attaching more weight to this than others. However, referring to literature, no particular method seems to have been developed for the laboratory granulation test. This has given the initiative for testing out several different methods in this laboratory to find out which would give the most consistent results.

Experimental Part

All experiments were carried out on an Allis Experimental Reduction Machine, manufactured by the Allis-Chalmers Manufacturing Company, this being equipped with one pair of corrugated rolls, one pair of smooth rolls and a gyrating sifter (speed 196 r.p.m.), provided with a set of interchangeable sieves for making separations through different numbers of bolting cloth, varying in degree of fineness of mesh, numbers 10XX to 16XX inclusive. The latter is, with occasional exceptions, the finest sieve with which the majority of mills is equipped.

As most American testing laboratories are equipped with this type of an experimental mill, the methods used should prove applicable.

The same flour was used for all experiments—a bleached second patent bakers' flour from 1927 crop Western Canada wheat. Chemical analysis of this flour is shown in Table I. The same amount of flour (100 grams) was used in all tests. Experiments were made with different amounts of flour, but 100 grams seemed best suited to this type of sieve.

The work was undertaken with the following problems in mind:

1. To find the variation in results caused by altering the manipulation and time.
2. To determine the influence of different degrees of temperature and relative humidity, as well as their combined effect.

3. To find to what extent the moisture content of flour influences results.
4. To ascertain any variation in results caused by the presence of coarse granulated particles in the flour.
5. To find the influence of actual conditions of sieves.

In the preliminary tests made, no attempt was made to control temperature and humidity. Several methods were tried out, varying the manipulation and the time of sifting, the idea being to find the method which would give the most consistent results and which, at the same time, would not be too cumbersome to be practically employed in making the granulation test.

TABLE I

ANALYSIS OF FLOUR USED

	Moisture	Ash	Protein	Gluten		Quality of Gluten	Absorption	Remark
				Wet	Dry			
	%	%	%	%	%		%	
As received	12.30	0.48	11.81	36.25	12.05	very good	57.20	Traces of Cl, NO_3
Computed to 13.5% Moisture basis		0.47	11.65	35.75	11.89			

It was found that when the sieves were placed on top of one another, that is, when more than one sieve was used at a time, it was impossible to obtain reasonably close agreement between results of duplicate tests, owing to the fact that there seemed to be considerable variation in the rate and completeness with which the flour dropped from one sieve to another. In view of this, the remainder of the experiments were made using one sieve at a time on the sifter.

Tests were made where the sifting started with the coarsest mesh, 10XX, and was continued up to the finest mesh, namely 16XX, the coarse portion held up on the sieves being weighed and discarded each time. It was found that by so doing approximately 40% of the flour passed through whilst 60% was held up on the sieves. On the other hand when the method of procedure was reversed and sifting commenced with the finest mesh, 16XX, it was found that altogether 64% of the flour passed through and only 36% was held up on the sieves. In this case the flour which passed through the different sieves was the portion weighed and discarded. From this it seemed evident that the coarse particles of flour acted as a cleaner, thereby assisting in the thoroughness of sifting.

Owing to the consistent results obtained using sieve 16XX, it was decided to use this sieve alone in trying out different cleaners

as used in commercial practice, and in testing out different periods of time for sifting.

The following cleaners were used:

1. The commercial cleaner as used underneath the sieves in America.
2. Steel chains, as used on top of the old type commercial sifters in America, varying the length of the chains to allow for different play on the sieves.
3. In using a brush as a cleaner this was tried out on top of the sieves, although for this purpose in Middle Europe similar type of brushes are used underneath the sieves.

   None of these three cleaners proved satisfactory inasmuch as there were abnormal variations in the results of tests.
4. Different amounts of wheat were tried out and for the amount of flour used in these tests, namely 100 grams, the best results were obtained using 20 grams of wheat. In all experiments where wheat was used as a cleaner, Manitoba No. 1 Northern was used. With this method, not only did results of duplicate tests correspond very closely but the time of sifting was reduced by half. There would be no great variation in size or in the specific gravity of wheat within certain grades from year to year, especially in the higher grades, and any error caused by such variation would be almost negligible. Under the circumstances and owing to the fact that wheat is not a scarce commodity in the laboratory, there should be no objection to its use as a cleaner in making the granulation test. On the other hand wheat is used as a cleaner in commercial practice in different parts of Europe (Czechoslovakia, Hungary). In Canada and the United States, however, the sifters are of a different type, making this method more or less impractical in commercial mills. In all experiments referred to later in this paper, wheat was used as a cleaner.

To find how the time influenced results and to determine the best time of sifting, experiments were carried out as follows:

1. Continuous sifting for a period of 98 minutes during which time the flour that adhered to the bottom of the sieve was not removed, only that portion which dropped through being weighed at intervals of ½, 1½, 3½, 6½, 10½, 15½, 23, 33, 48, 68, and 98 minutes.
2. Experiments were also made varying the time of sifting as follows: ½, 1, 2, 3, 4, 5, 7½, 10, 15, 20, 30 and 60 minutes. Each

experiment was complete in itself as a new portion of 100 grams of flour was used each time. The bottom of the sieve was carefully brushed so that everything passing through was weighed.

Where sifting was continuous, although similar to commercial practice, it was obvious that the silks required cleaning as the condition of the flour adhering so closely to the bottom of the sieves influenced the time of sifting as well as the percentage of flour passing through. The results were more satisfactory when the bottom of the sieves was brushed and when a fresh amount of flour was used with each sieve. However, in further experiments both methods were utilized as it was felt that more information could thus be gained. To differentiate between these two methods, experiments are marked A where sifting was continuous as in the first method mentioned above, the second method being marked B.

In regard to the time of sifting, between 70% and 80% of the flour passed through the sieves in 15½ minutes, which fact would indicate that fairly thorough separation of the particles of flour took place within this time, as only a small percentage passed through with continued agitation. In view of this, the time limit of 15½ minutes sifting was adopted for further tests. This should not prove impractical in making the laboratory granulation test.

To find any changes in the moisture content during sifting, tests were made at regular intervals. With flour having a moisture content of 12.30%, the portion passing through 16XX had dried out to 10.30% at the end of 20 minutes sifting, and when sifting was continued for 30 minutes it had dried out to 9.20%. At this time the moisture content of the coarse particles of flour still held up on the sieves was 8.30%.

Whilst it was possible to get close results in duplicate tests made directly after one another, there was a variation if any length of time intervened, and the difference in results was quite marked if the tests were run on different days. Observing the rise and fall of the invisible loss or gain which occurred in yield from day to day led up to experiments being made to *find the extent of the influence on sifting and on yield of changes in atmospheric conditions. To this* end, experiments were made to determine the influence on sifting of varying degrees of relative humidity, and of temperature, as well as their combined influence, each of these factors being kept under control.

A steam joint was used in the room for direct humidifying and the relative humidity was determined on an autometer or a wet

and dry bulb thermometer, the accuracy of this being checked periodically with a sling psychrometer. To regulate stated periods of sifting, an automatic stop-watch recording seconds was used.

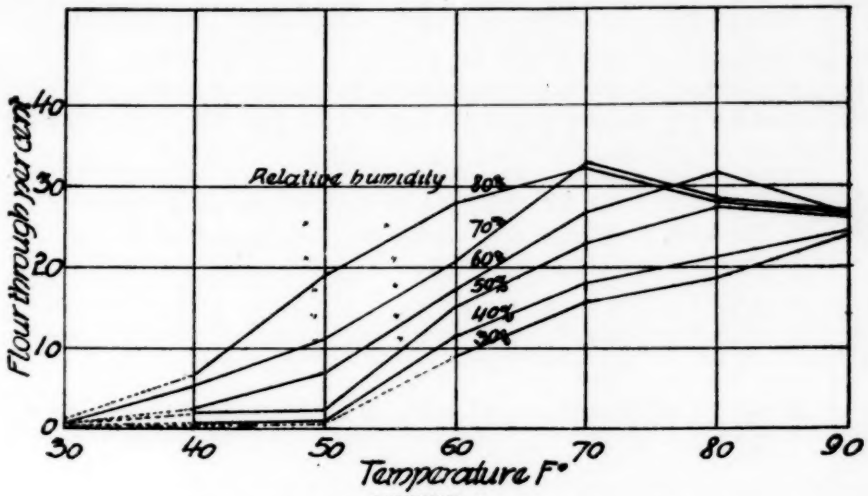
There was no great difficulty in regulating the temperature of the flour but it was not such an easy matter to control room temperature and relative humidity. There were apt to be variations in either direction, so that allowance must be made for an error of $\pm 1°$ in room temperature and $\pm 2\%$ in relative humidity. This is especially necessary with room temperature and relative humidity in the high or low extremes.

In the experiments made, the temperature of the room and of the flour ranged between 30°F. (—1.1°C.) and 90°F. (32.2°C.) and the relative humidity between 30% and 80%, this being the highest degree which could be obtained under laboratory conditions. It was impossible to obtain a relative humidity of 30% with a temperature of 40° and 50°F. (4.4° and 10.0°C.) and with the temperature as low as 30°F. (—1.1°C.) the relative humidity could not be regulated at all. The only test which could be made was with 70% relative humidity, this being the humidity which was present in the air with a temperature of 30°F. (—1.1°C.). Results of these tests are shown in Tables II and III.

As the rate of sifting in commercial practice is an economic factor, the amount of flour passing through in initial sifting, or in the first moment the flour strikes the sieve, is an important consideration. It is interesting to know to what extent temperature and relative humidity influence the results that can be obtained in such a short space of time (Figs. 1 and 2). There was practically no sifting at freezing point when the actual percentage of the moisture content of the air was very low, and with low temperatures 30°, 40°, and 50°F. (—1.1°, 4.4°, and 10.0°C.), and low relative humidity 30%, 40%, 50%, very little flour was passing through. The flour appeared to be absolutely lacking in liveliness and the mesh of the silk became clogged. This was particularly noticeable with Method A, in which the bottoms of the sieves were not brushed.

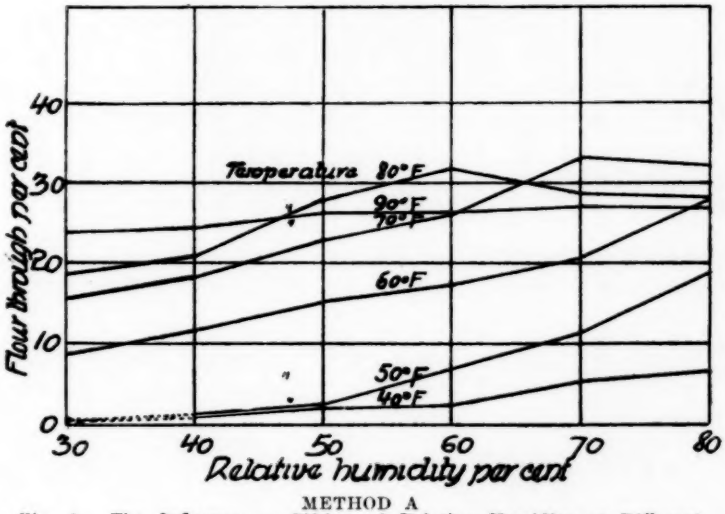
With relative humidity at 60%, 70% and especially 80%, even with the temperature at 40°F. (4.4°C.), it influenced the rate of sifting and there was an increase in the amount passing through during the first half minute. This was still more pronounced at 50° and 60°F. (10.0° and 15.6°C.). At 70°F. (21.1°C.) the peak is reached where 70% relative humidity influences sifting to a greater extent than does 80% relative humidity. The next best results

were obtained with 60% relative humidity and 80°F. (26.7°C.). These peaks are clearly shown in Figs. 1 and 2. With relative hu-



METHOD A

Fig. 1. The Influence on Sifting of Temperature at Different Relative Humidities. Time of Sifting ½ Min.



METHOD A

Fig. 2. The Influence on Sifting of Relative Humidity at Different Temperatures. Time of Sifting ½ Min.

midity above 60% and the temperature above 80°F. (26.7°C.), there is a decrease in the amount of flour passing through. At 90°F. (32.2°C.) when the actual moisture content in the air is very high, increasing relative humidity has very little influence on sifting during the first half minute.

TABLE II

COMBINED INFLUENCE ON SIFTING OF RELATIVE HUMIDITY AND TEMPERATURE
Method "A" — Continuous Sifting

Temperature of Room	Temperature of Flour	Relative Humidity	Time of Sifting in Minutes ½	1½	3½	6½	10½	15½	Total Passing Through	Total Held up on Sieve	Sum Total	Invisible loss— gain +
° F	° F	%	%	%	%	%	%	%	%	%	%	%
30	30	70	0.32	1.76	14.67	37.73	13.46	6.18	74.12	25.52	99.64	—0.36
		40	0.65	5.30	13.93	20.40	22.90	12.00	75.18	21.77	96.95	—3.05
		50	1.90	11.95	30.38	21.75	6.91	4.28	77.17	21.63	98.80	—1.20
40	40	60	2.30	14.96	39.37	12.64	5.66	3.47	78.40	20.87	99.27	—0.73
		70	5.16	15.18	37.66	12.30	5.05	3.59	78.94	21.38	100.32	+0.32
		80	6.20	35.41	24.40	7.70	4.37	2.56	80.64	20.05	100.69	+0.69
		40	0.85	6.30	15.36	21.50	20.84	11.60	76.45	21.18	97.63	—2.37
		50	2.02	19.15	35.92	13.29	5.27	3.55	79.20	19.76	98.96	—1.04
50	50	60	6.63	34.02	25.49	6.96	3.79	3.35	80.24	19.33	99.57	—0.43
		70	11.12	38.85	17.44	5.80	3.60	2.18	78.99	21.19	100.18	+0.18
		80	18.97	40.98	9.55	3.69	2.48	2.23	77.90	23.23	101.13	+1.13
		30	8.65	32.01	25.07	7.08	3.87	3.12	79.80	17.79	97.59	—2.41
		40	11.59	34.78	19.90	6.41	3.85	2.81	79.34	18.86	98.20	—1.80
		50	15.07	36.29	15.83	5.86	3.23	2.77	79.05	19.71	98.76	—1.24
60	60	60	16.95	38.40	13.15	5.13	3.12	2.65	79.40	20.50	99.90	—0.10
		70	20.35	37.13	10.53	4.37	2.96	2.14	77.48	23.09	100.57	+0.57
		80	27.82	30.98	8.33	3.71	2.64	1.97	75.45	25.71	101.16	+1.16
		30	15.61	35.11	17.25	5.81	3.59	2.90	80.27	16.98	97.25	—2.75
		40	18.15	34.37	14.94	5.44	3.50	2.80	79.20	19.73	98.93	—1.07
		50	22.70	33.63	12.64	5.08	3.78	2.70	80.53	18.47	99.00	—1.00
70	70	60	26.78	31.54	10.52	4.38	3.03	2.26	78.51	21.31	99.82	—0.18
		70	32.86	29.45	8.41	3.68	2.28	1.82	78.50	21.81	100.31	+0.31
		80	32.05	25.73	7.95	3.76	2.38	2.13	74.00	27.11	101.11	+1.11
		30	18.27	35.68	14.18	5.53	3.21	2.98	79.85	16.60	96.45	—3.55
		40	20.83	32.45	13.30	5.53	3.36	2.32	77.79	20.55	98.34	—1.66
		50	27.52	26.82	10.78	5.20	3.40	2.63	76.35	23.16	99.51	—0.49
80	80	60	31.72	23.68	8.65	4.53	3.11	2.73	74.42	25.58	100.00	±0.00
		70	28.32	23.00	9.75	4.18	3.61	2.86	71.72	28.89	100.61	+0.61
		80	28.00	22.20	9.55	5.07	3.54	2.98	71.34	30.00	101.34	+1.34
		30	23.93	31.62	12.50	5.16	3.43	2.80	79.44	15.74	95.18	—4.82
		40	23.34	28.69	12.60	6.75	4.02	2.80	79.20	19.95	99.15	—0.85
		50	26.02	27.13	11.72	5.67	3.49	2.44	76.47	23.14	99.61	—0.39
90	90	60	26.46	24.72	10.21	4.98	3.44	3.16	72.97	27.55	100.52	+0.52
		70	26.70	22.96	10.72	5.52	3.86	2.96	72.72	28.59	101.31	+1.31
		80	26.46	22.79	10.58	5.51	3.73	2.97	72.04	29.53	101.57	+1.57

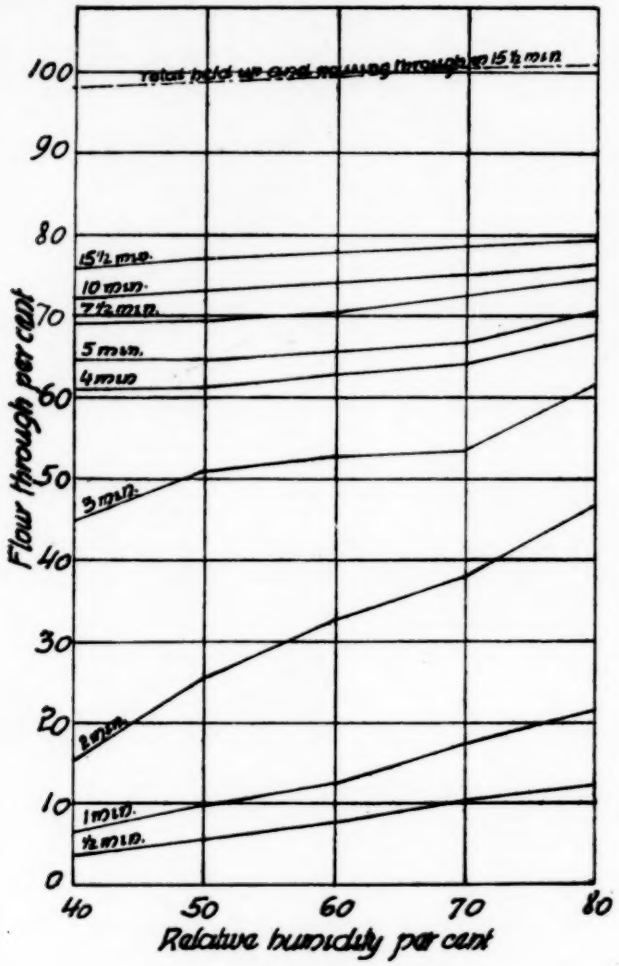
		80	26.46	22.79	10.58	5.51	3.73	2.97	72.04	29.53	101.57	+1.57

TABLE III

Method "B" — New Portion of Flour Used for Each Sifting

Temperature of Room	Temperature of Flour	Relative Humidity	Time of Sifting in Minutes ½	1	2	3	4	5	7½	10	15½	Held up on Sieve After 15½ min.	Total Held up and Passing Through in 15½ min.	Invisible Loss — or Gain +
° F	° F	%	%	%	%	%	%	%	%	%	%	%	%	%
30	30	70	1.50	3.94	6.68	9.58	24.78	43.22	58.65	66.12	73.57	25.13	98.70	—1.30
		40	3.80	6.60	15.77	44.76	60.98	64.58	69.18	71.23	75.95	22.20	98.15	—1.85
		50	5.77	9.63	25.53	51.20	61.28	64.83	69.66	72.97	77.12	21.81	98.93	—1.07
40	40	60	7.74	12.48	32.69	52.82	62.96	65.53	70.65	73.90	78.06	21.48	99.54	—0.46
		70	10.11	17.33	37.72	53.52	64.02	66.60	72.50	74.87	78.74	21.67	100.41	+0.41
		80	12.45	21.34	46.45	61.88	67.86	70.32	74.47	76.39	79.67	21.01	100.68	+0.68
		40	4.61	7.44	23.58	50.02	61.93	65.46	71.77	75.13	78.55	17.98	98.33	—1.67
		50	8.10	19.84	41.20	60.67	66.34	69.75	73.85	76.10	78.90	20.11	99.01	—0.99
50	50	60	13.06	26.63	53.31	63.21	68.14	70.75	74.30	76.03	79.92	19.76	99.68	—0.32
		70	16.20	35.35	58.85	65.49	68.70	70.30	74.18	75.90	78.52	21.66	100.18	+0.18
		80	25.58	49.87	62.40	66.61	69.01	69.95	73.01	75.08	77.27	23.72	100.99	+0.99
		30	11.50	22.75	53.81	62.70	67.89	69.50	73.85	75.46	79.06	19.45	98.51	—1.49
		40	15.51	29.01	55.98	64.20	68.71	70.50	74.85	77.17	80.00	18.92	98.92	—1.08
60	60	50	19.28	35.27	58.16	65.15	69.61	70.30	74.73	76.74	79.50	19.87	99.37	—0.63
		60	21.75	45.67	62.01	67.27	70.51	71.10	73.95	76.40	79.25	20.63	99.88	—0.12
		70	29.74	51.77	64.25	68.67	70.26	70.86	72.07	74.58	77.12	23.39	100.51	+0.51
		80	31.25	49.95	61.44	65.33	67.67	68.18	71.63	72.75	75.23	25.73	100.96	+0.96
		30	15.82	36.25	58.53	65.75	70.47	72.44	74.56	76.93	80.87	17.43	98.30	—1.70
		40	19.87	40.89	60.00	66.87	70.26	72.36	74.00	76.29	78.50	20.58	99.08	—0.92
70	70	50	22.70	44.62	61.82	68.00	70.05	72.28	73.51	75.31	77.68	21.72	99.40	—0.60
		60	28.86	49.43	62.21	66.96	69.70	71.59	73.01	74.33	76.87	23.08	99.95	—0.05
		70	35.46	54.31	63.86	66.53	69.18	70.20	72.96	73.62	75.89	24.64	100.53	+0.53
		80	33.20	51.65	60.71	64.84	65.99	67.57	68.98	71.17	73.16	28.05	101.21	+1.21
		30	23.04	48.32	63.74	67.40	69.95	71.70	75.14	76.67	78.93	19.30	98.23	—1.77
		40	26.62	47.22	61.07	66.27	68.80	70.90	72.82	74.90	77.57	21.91	99.48	—0.52
80	80	50	28.28	46.00	59.55	64.27	67.87	69.06	71.83	73.20	76.22	23.61	99.83	—0.17
		60	31.66	45.80	58.99	62.34	64.91	66.52	69.66	71.68	74.35	26.24	100.59	+0.59
		70	30.40	44.63	56.71	61.73	63.29	64.84	67.70	69.62	73.01	27.74	100.75	—0.75
		80	30.00	44.50	55.70	61.13	62.70	64.72	67.11	69.26	71.21	28.98	101.98	+1.19
		30	25.81	47.54	61.41	66.05	69.02	71.05	74.64	76.53	79.44	19.27	98.71	—1.29
		40	26.02	45.69	59.80	64.98	68.18	69.83	73.23	75.59	78.25	21.22	99.47	—0.53
90	90	50	26.23	43.84	57.19	63.02	65.57	67.07	69.83	72.13	76.01	23.76	99.77	—0.23
		60	26.54	42.58	54.03	59.89	62.11	64.74	67.48	70.05	73.08	27.70	100.78	+0.78
		70	27.85	42.06	53.87	59.29	61.54	64.08	67.09	69.52	72.69	28.67	101.36	+1.36
		80	27.93	41.13	53.48	58.70	60.98	63.23	66.67	68.80	72.30	29.12	101.42	+1.42

Comparing charts represented by Figs. 1 and 2, it is apparent that *temperature, especially above 50°F. (10.0°C.) has a greater influence on sifting during the first half minute than relative humidity.* At the same time it is interesting to follow the combined influence of the two factors.

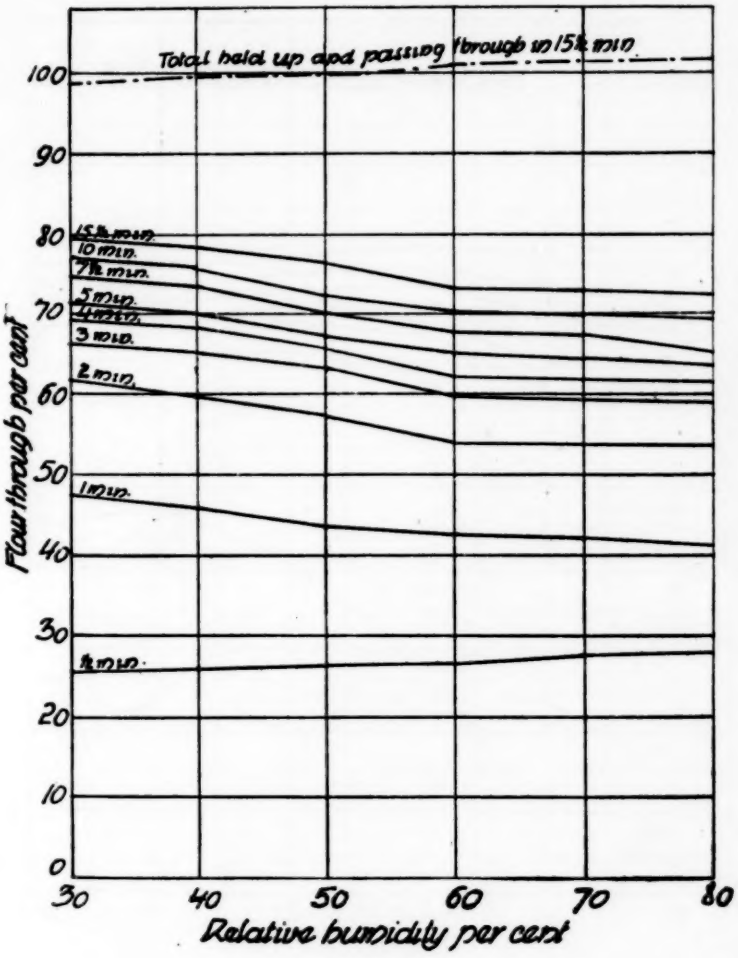


METHOD B

Fig. 3. The Influence on Sifting of Different Relative Humidities with Temperature of Room and Flour at 40° F. Invisible Loss or Gain Illustrated

As regards the continued influence of temperature and relative humidity over longer periods of sifting: With increasing relative humidity, and a temperature of 40°F. (4.4°C.), there is an increase in the amount of flour passing through at any period of time up to

15½ minutes. (Fig. 3.) With the temperature at 50°F. (10.0°C.), increasing relative humidity influences increase in sifting only up to a period of four minutes, after which time with more than 60% relative humidity, there is a decrease in the amount of flour passing through in 15½ minutes. With increasing high temperature at 60°, 70° and 80°F. (15.6°, 21.1° and 26.7°C.) and as the relative



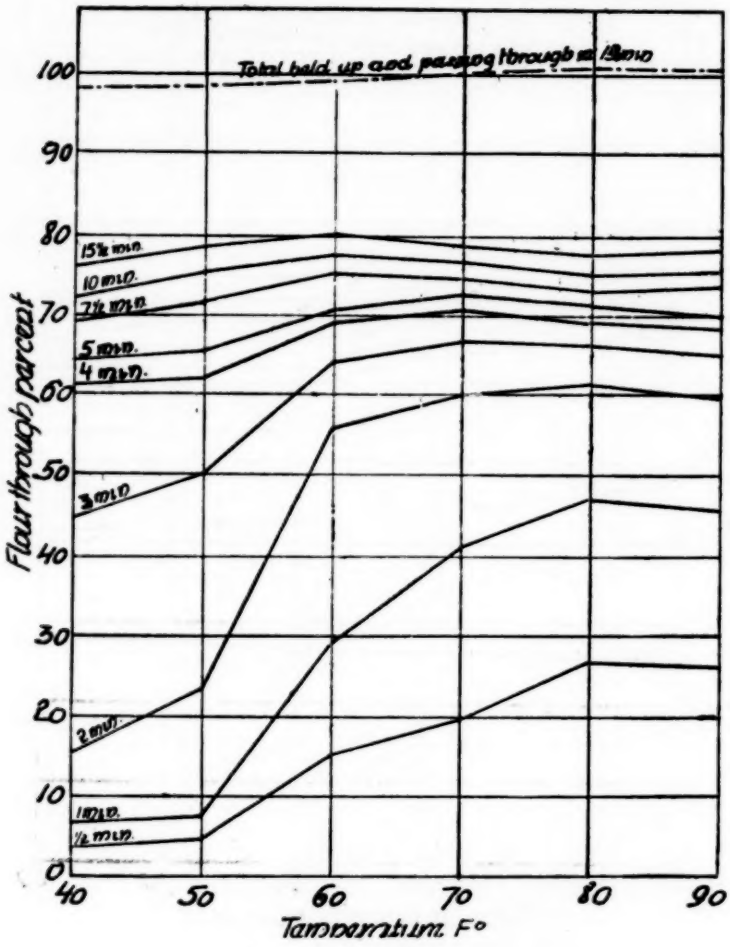
METHOD B

Fig. 4. The Influence on Sifting of Different Relative Humidity with Temperature of Room and Flour at 90° F. Invisible Loss or Gain Illustrated.

humidity is increased, there is a marked decrease in the results of sifting over a period of 15½ minutes. This tendency to show a decrease in sifting is still more pronounced with the temperature at

90°F. or 32.2°C. (Fig. 4.) With temperatures at 80° and 90°F. (26.7°C. and 32.2°C.) the only increase shown in sifting was during the first half minute, and in the case of 90°F. or 32.2°C. this increase was only a very slight one.

Figs. 3 and 4 show mainly the influence on sifting, over a period of 15½ minutes, of different relative humidities when the

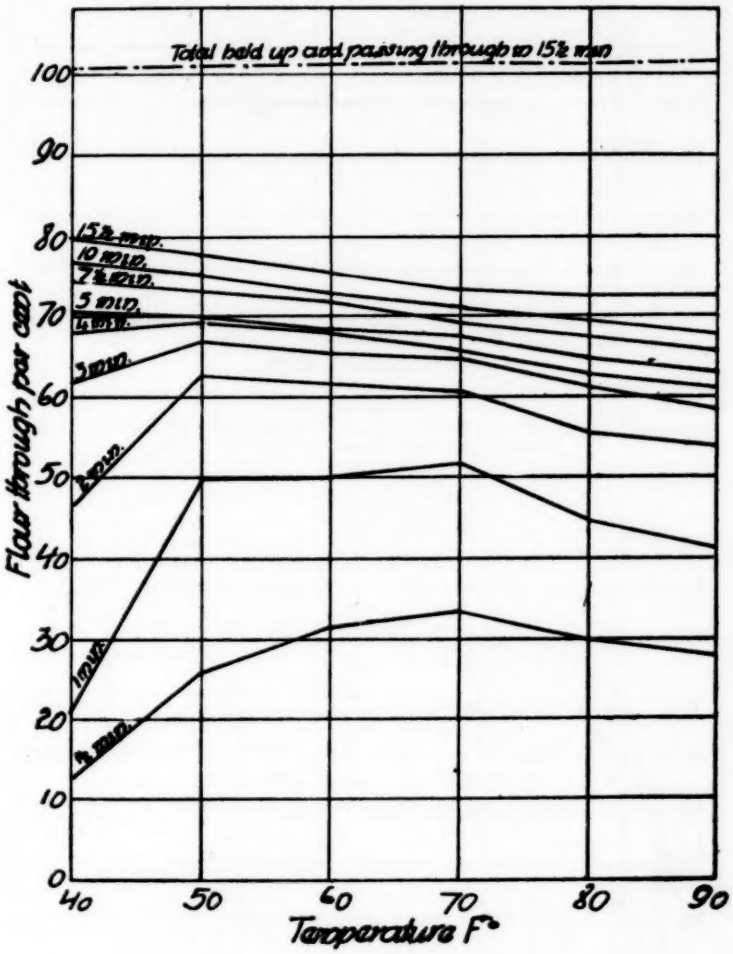


METHOD B

Fig. 5. The Influence on Sifting of Different Temperatures with 40% Relative Humidity. Invisible Loss or Gain is Illustrated

the influence of different temperatures with relative humidity at 40% and 80%. These charts are based on results obtained using temperature is in the low and high extreme. Figs. 5 and 6 illustrate

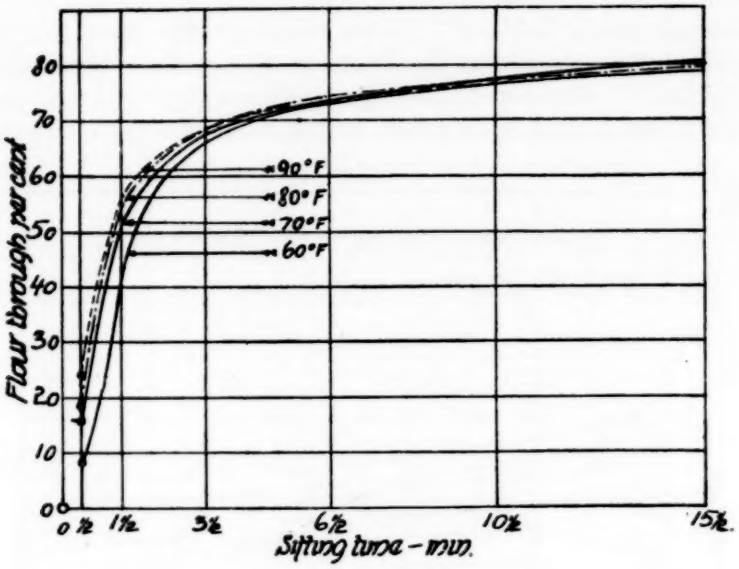
Method B, indicating the amount of flour passing through for given periods of time. Table III (from which graphs may be made) shows results obtained at intervening temperatures and relative humidities. Under corresponding conditions Table II shows the results from Method A, in which only one lot of flour (100 grams)



METHOD B

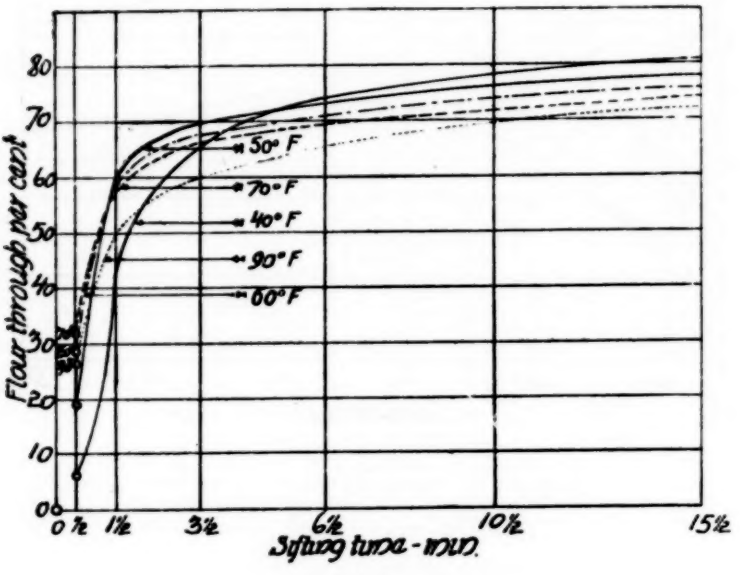
Fig. 6. The Influence on Sifting of Different Temperatures with 80% Relative Humidity. Invisible Loss or Gain is Illustrated

was used and sifting was continuous over a period of 15½ minutes. The figures in Table II denote only the amount of flour passing through in the intervals between stated times.



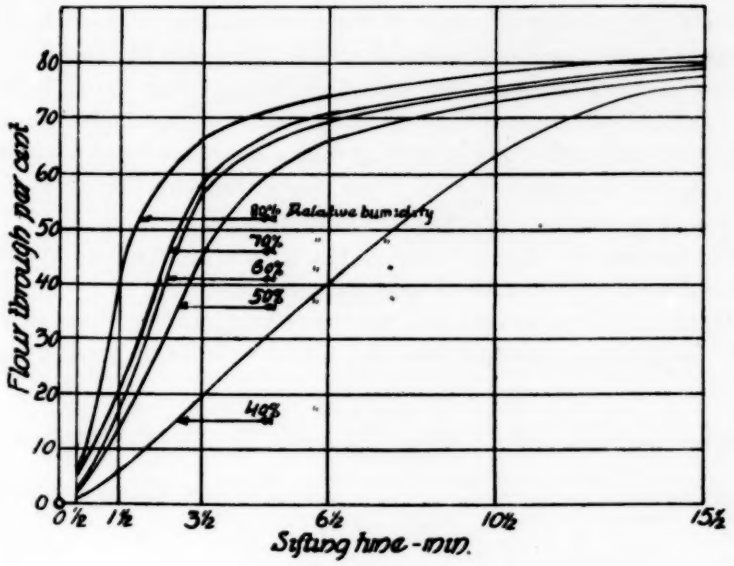
METHOD A

Fig. 7. The Influence on the Intensity of Sifting of Different Temperatures. Relative Humidity 30%



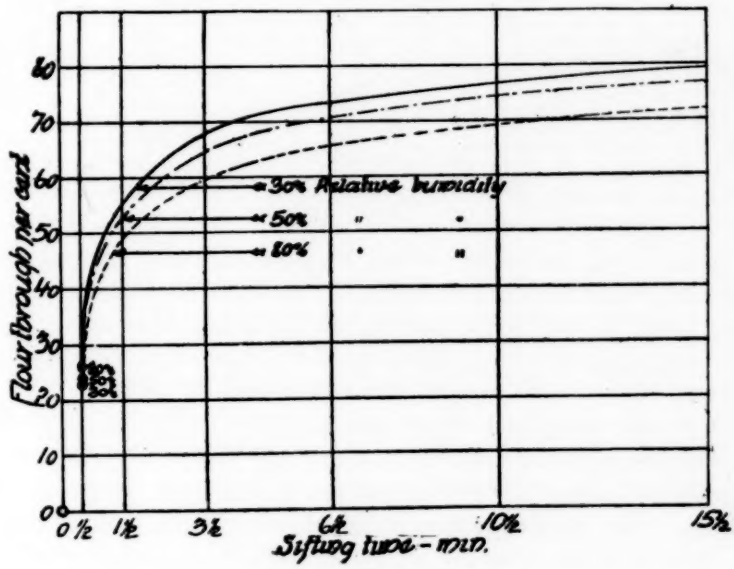
METHOD A

Fig. 8. The Influence on the Intensity of Sifting of Different Temperatures. Relative Humidity 80%



METHOD A

Fig. 9. The Influence on the Intensity of Sifting of Different Relative Humidities. Temperature of Room and Flour 40° F.



METHOD A

Fig. 10. The Influence on the Intensity of Sifting of Different Relative Humidities. Temperature of Room and Flour 90° F.

On the other hand Figs. 7, 8, 9 and 10 are based on Method A, but the flour passing through in each succeeding interval is added, so that the lines on these charts actually represent the total amount of flour passing through for stated periods. From these it is evident that different conditions of temperature and relative humidity cause extreme variations in sifting and a decrease over a period of 15½ minutes is influenced to a greater extent by increasing relative humidity than by increasing temperature.

Comparing Methods A and B there is very little difference in the total yield at the end of 15½ minutes.

With low temperature and low relative humidity there is a tendency for the flour to dust into the air. At the same time evapora-

TABLE IV

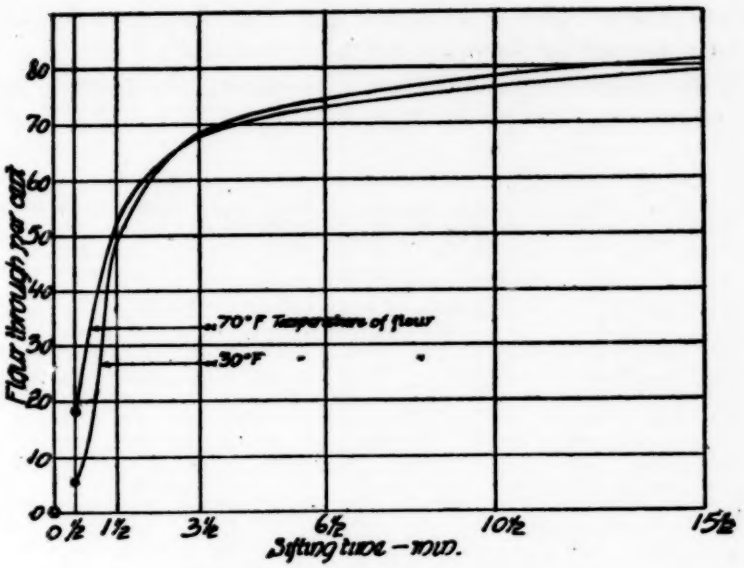
COMBINED INFLUENCE ON SIFTING OF DIFFERENT TEMPERATURES OF ROOM AND FLOUR AT 40% RELATIVE HUMIDITY

Method "A" — Continuous Sifting

Time of Sifting in Minutes	Temperature of room ° F.	70		40			
	Temperature of flour ° F.	30	70	40	75	110	140
				Flour passing through %			
½		5.19	18.15	0.65	23.00	27.75	37.67
½— 1½		43.13	34.37	5.30	27.70	20.80	19.71
1½— 3½		19.58	14.94	13.93	13.44	13.30	8.34
3½— 6½		6.24	5.44	20.40	6.27	6.74	5.85
6½—10½		3.67	3.50	22.90	3.72	4.18	3.71
10½—15½		2.71	2.80	12.00	3.17	3.74	3.41
Total passng through %		80.52	79.20	75.18	77.30	76.51	78.69
Total held up on sieve, %		17.44	19.73	21.77	17.08	17.46	15.41
Sum total, %		97.96	98.93	96.95	94.38	93.97	94.10

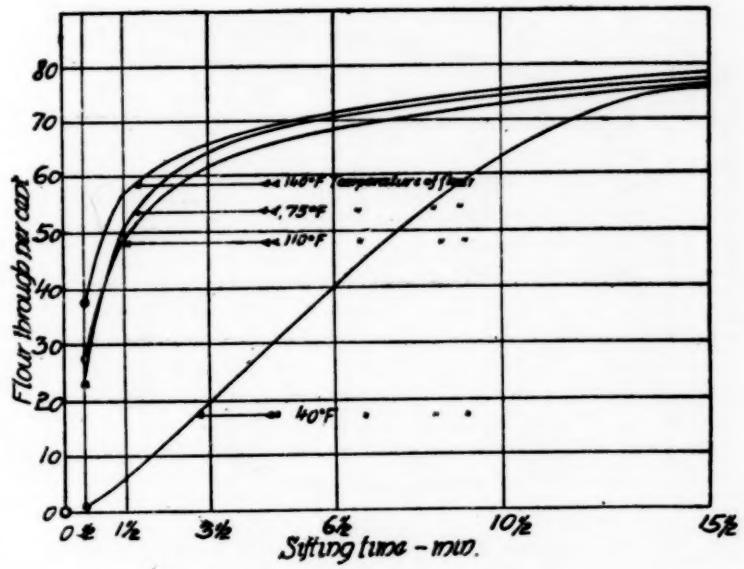
tion of the moisture of the flour takes place, this evaporation being greater when the moisture content of the flour is high. These conditions are responsible for a loss in yield. On the contrary, with increasing temperature and especially with increasing relative humidity there is very little dusting of the flour. Moreover, instead of any evaporation taking place, the flour takes up additional moisture from the atmosphere, resulting in a gain in yield.

In all the foregoing experiments the temperature of the flour and of the room were kept the same. Figs. 11 and 12 illustrate variations in the results of sifting caused by extreme temperatures in the flour. One experiment was made with room temperature at 40°F. (4.4°C.) and another at 70°F. (21.1°C.) and in each case flours with different temperatures were used. From the results obtained (Table IV) it is evident that the temperature of the flour



METHOD A

Fig. 11. The Influence on Intensity of Sifting of Low Temperature in Flour (30° F.) as Compared with 70° F. Room at 70° F. and 40% Relative Humidity.



METHOD A

Fig. 12. The Influence on Intensity of Sifting of Extreme Temperatures in Flour with Temperature of Room at 40° F. and 40% Relative Humidity

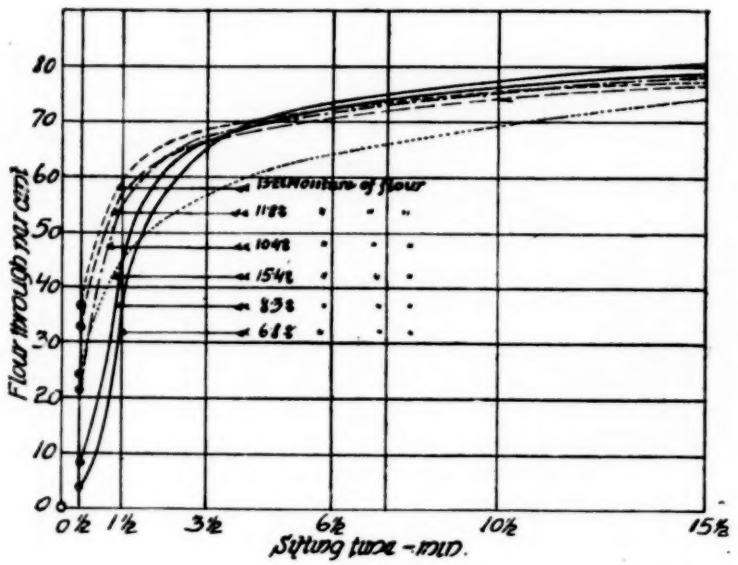
itself influences the rate at which the flour passes through the sieve, particularly in the initial sifting.

To find the influence on sifting of the moisture content in flour, experiments were made with flours in which the moisture content

TABLE V

THE INFLUENCE OF SIFTING OF DIFFERENT MOISTURES IN FLOUR WITH TEMPERATURE OF ROOM AND FLOUR AT 70° F. AND 70% RELATIVE HUMIDITY
Method "A" — Continuous Sifting

Time of Sifting in Minutes	Moisture Content %									
	6.80	8.30	9.30	10.40	11.40	11.80	12.30	13.20	14.20	15.40
	Flour Passing Through %									
½	3.41	8.14	15.08	21.26	28.77	32.71	35.46	36.71	29.29	24.33
½— 1½	31.37	36.38	36.34	33.53	27.70	23.90	24.14	22.45	21.10	20.60
1½— 3½	30.13	21.45	16.11	11.73	9.91	9.05	8.61	8.93	10.72	11.92
3½— 6½	7.92	6.30	5.21	5.26	4.84	4.81	3.93	4.21	6.93	7.04
6½—10½	4.31	3.55	3.52	3.44	3.61	3.25	2.75	2.83	5.24	5.27
10½—15½	3.24	2.88	2.68	2.57	2.78	2.65	2.08	2.16	3.64	4.60
Total passing through %	80.38	78.70	78.94	77.79	77.61	76.37	75.97	77.29	76.92	73.76
Total held up on sieve %	20.44	21.08	20.41	21.00	21.14	22.60	21.91	21.56	20.99	23.85
Sum total %	100.82	99.78	99.35	98.79	98.75	98.97	98.88	98.85	97.91	97.61



METHOD A

Fig. 13. The Influence on Intensity of Sifting of Different Moistures in Flour with Room at 70° F. and 70% Relative Humidity

ranged between 6.80% and 15.40%, the factors of temperature 70°F. (21.1°C.) and relative humidity 70% being kept constant.

TABLE VI

INFLUENCE ON SIFTING OF COARSE GRANULATED PARTICLES IN FLOUR
Method "A" — Continuous Sifting

Time of Sifting in Minutes	Flour No. 1	Flour No. 2	Flour No. 3	Flour No. 4	Flour No. 5	Flour No. 6	Flour No. 7
				Passing Through %			
½	15.40	18.42	23.41	24.77	25.88	26.63	32.86
½— 1½	42.25	50.03	49.79	44.93	39.89	36.85	29.45
1½— 3½	39.53	28.54	20.37	16.24	14.87	10.39	8.41
3½— 6½	0.57	1.17	3.86	6.98	5.77	4.78	4.68
6½—10½			0.85	3.25	2.79	3.32	2.28
10½—15½				1.51	1.90	2.32	1.82
Total passing through %	97.75	98.16	98.28	97.68	91.10	84.29	78.50
Total held up on sieve %	0.32	0.49	0.42	1.11	8.15	15.19	21.81
Sum total %	98.07	98.65	98.70	98.79	99.25	99.48	100.31

Explanation re flours used in above experiments:

Normal flour was sifted ½ minute, in which time 32.86% passed through. This operation was repeated until 100 grams of such flour was obtained. This is designated as FLOUR NO. 1.

FLOUR NO 2 Composed of portions of 51.22% sifted through in 1 minute.

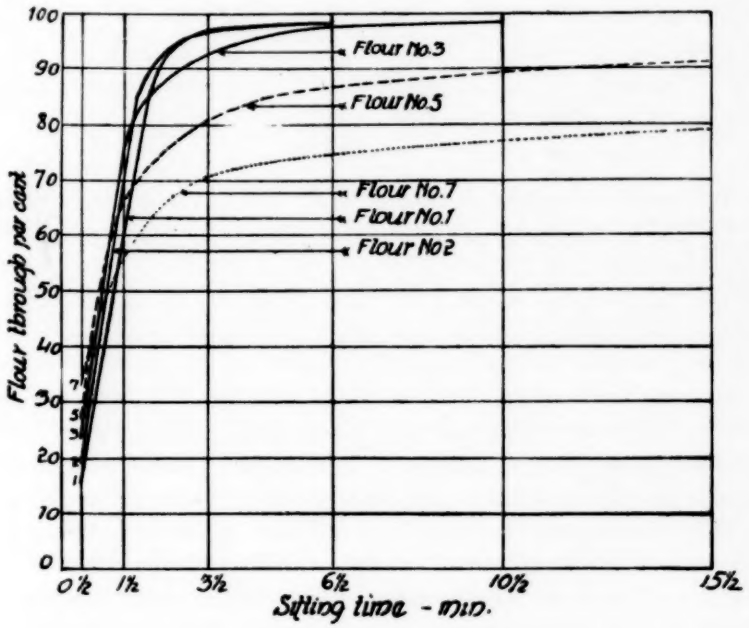
FLOUR No. 3 Composed of portions of 61.43% sifted through in 2 minutes.

FLOUR No. 4 Composed of portions of 71.94% sifted through in 3½ minutes.

FLOUR No. 5 Composed of portions of 71.94% sifted through in 3½ minutes plus portions of 8.06% of the flour held up on the sieve.

FLOUR No. 6 Composed of portions of 71.94% sifted through in 6 minutes plus portions of 18.06% of the flour held up on sieve.

FLOUR No. 7 Normal untreated flour.



METHOD A

Fig. 14. The Influence on Intensity of Sifting of Coarse Granulated Particles in Flour

Some of the flour was allowed to dry in the air, the moisture content of this flour then being 6.80%. To obtain the desired moisture content, portions of this air-dried flour were then blended with normal untreated flour having a moisture content of 12.30%. When it was necessary to have more than 12.30% moisture, the flour was placed in air saturated with moisture until it absorbed the additional moisture desired.

In the initial sifting the best results were obtained from the flour with 13.20% moisture (Table V and Fig. 13). All the flours with moisture in the high extremes passed through the silk much more quickly than the flours with low moisture but the reverse of this was true in sifting over a period of 15½ minutes.

TABLE VII

INFLUENCE ON RESULTS OF SIFTING OF ACTUAL CONDITIONS OF SIEVES—TEMPERATURE OF ROOM AND FLOUR 70° F. RELATIVE HUMIDITY 70%

Method "A" — Continuous Sifting

% moisture of flours used in experiments below		13.20			11.80		9.30		6.80	
After sifting flours containing moistures %	Normal Conditions[1]	11.80	15.40	13.20	13.20	6.80	10.40	9.30	13.20	Normal Conditions
Time of sifting in minutes	Passing Through %									
½	36.71	32.22	25.46	30.48	18.77	32.71	13.67	15.08	3.41	4.47
½— 1½	22.45	22.85	19.55	17.53	37.53	23.90	36.87	36.34	31.37	36.87
1½— 3½	8.93	10.75	9.51	9.65	12.00	9.05	16.10	16.11	30.13	25.86
3½— 6½	4.21	6.39	7.01	5.88	5.28	4.81	5.60	5.21	7.92	6.53
6½—10½	2.83	4.25	4.80	4.16	3.30	3.25	3.51	3.52	4.31	3.36
10½—15½	2.16	3.08	4.24	3.41	2.64	2.65	2.68	2.68	3.24	2.76
Total passing through %	77.29	79.54	70.57	71.11	79.52	76.37	78.43	78.94	80.38	79.85
Total held up on sieve %	21.56	18.98	27.87	28.03	18.00	22.60	20.51	20.41	20.44	21.42
Sum total	98.85	98.52	98.44	99.14	97.52	98.97	98.94	99.35	100.82	101.27

[1] Under "normal conditions" no flour is sifted before the experiment.

Experiments were carried out to find the influence on sifting of coarse granulated particles in flour (Table VI and Fig. 14).

With flour No. 1, which is composed of flour which passed through in ½ minute, it would be natural to expect that in re-sifting, this would pass through very quickly. However, this was not the case and in the first half-minute re-sifting, only 15.42% of the flour passed through as compared with 32.86% of the normal flour. There was a gradual increase in the amount passing through as the flours contained increasing percentages of the normal flour. On the other hand, sifting extended over a longer period of time than

with the flours containing a smaller percentage of the coarse particles. From this it is evident that the coarse particles influenced the rate at which the flour passed through the sieve.

The point was brought up as to whether or not actual conditions of the sieves might cause variations in results. To determine this, experiments were carried out using flours with different moisture contents. To begin with, an experiment was repeated four times with flour having 13.20% moisture, but before each experiment, a flour with an entirely different moisture was put through the sieve. In a like manner, other experiments were made with flours having a moisture content of 11.80%, 9.30%, and 6.80%. From the figures shown in Table VII it is clear that different conditions of moisture have a temporary effect on the sieves and that these in turn have a similar influence on the results of sifting. It would therefore be impossible to obtain any agreement between results unless duplicate tests were made directly after one another.

Under controlled conditions of temperature 70°F. (21.1°C.) and relative humidity 70% several commercial flours were tested using different silks and it was found that between 50% and 60% of each flour passed through silk No. 25 Standard in 15 minutes and when sifting was continued with silks Nos. 20 Standard and 16XX and 13XX, all except 2% to 4% of the remaining flour passed through in a further period of 15 minutes.

Conclusions

The method of using one sieve at a time was adopted, as it was impossible to obtain consistent results when the sieves were placed on top of one another, owing to the variations in the rate and completeness with which the flour dropped from one sieve to another.

When sifting commenced with No. 16XX, instead of with a coarse mesh, there was an increase in the amount of flour passing through the sieves.

Out of four different types of cleaners used, wheat proved the most satisfactory. Not only did the results of duplicate tests correspond very closely, but with wheat as a cleaner, the time of sifting was reduced by half.

In experiments where sifting was continued over a long period of time it was found that between 70% and 80% of the flour passed through the sieve in 15 minutes, this fact indicating that fairly thorough separation of the particles took place within this time.

When sifting was continuous and the bottoms of the sieves

were not brushed, these conditions had a retarding effect on sifting. However, comparing results obtained using Methods A and B there is very little difference in the total amount of flour passing through over a period of 15½ minutes.

As sifting proceeded over a period of 30 minutes the flour gradually dried out, thereby producing changes in the moisture content. These results were obtained under existing conditions of temperature and relative humidity. When these factors were controlled it was evident that flour with a high moisture content dried out more during sifting than flour with less moisture. In other words, with increased percentages of moisture in the flour greater evaporation took place during sifting, resulting in a corresponding loss in yield. In the initial sifting the best results were obtained from flour with 13.20% moisture and all the flours with moisture in the high extremes passed through the sieve much more quickly than the flours with moisture in the other extreme. The reverse of this was true in sifting over a period of 15½ minutes.

Under controlled atmospheric conditions, with low temperatures 30°, 40° and 50°F. (—1.1°, 4.4° and 10.0°C.) and low relative humidities, 30%, 40% and 50%, the flour lacks liveliness and the mesh of the silk becomes clogged up; consequently only a small percentage of flour passed through during the first half minute. However, when either one of these factors was increased, irrespective of the other, it affected an increase in sifting up to a certain point. Temperature especially above 50°F. (10.0°C.), has a greater influence on initial sifting than has relative humidity. The best results were obtained with temperature of room and flour at 70°F. (21.1°C.) and 70% relative humidity.

Over a period of 15½ minutes different conditions of temperature and relative humidity were responsible for wide variations in the results of sifting. Any tendency towards a decrease in the amount of flour which passed through the sieve was influenced to a greater extent by increasing relative humidity than by increasing temperature.

Extreme temperatures in the flour influence results which can be obtained, particularly in initial sifting.

During sifting, with conditions of low temperature and low relative humidity, the flour dusts into the air. The loss thus sustained, accompanied by the loss due to the flour drying out, is responsible for low yield. On the contrary, with increasing temperature and more especially with increasing relative humidity, there

is very little dusting and the flour itself takes up additional moisture from the atmosphere, resulting in higher yield.

Coarse particles in flour, in proportion to the percentage in which these are present, are responsible for increases in the amount of flour passing through the sieves.

Allowances should be made for possible errors due to conditions of the equipment and the quality of the silks used, as well as to the method itself. Results of experiments proved that different conditions of temperature, relative humidity, and the actual moisture content of the flour all had a specific effect on the sieves. The sieves, thus temporarily effected, had in turn an influence on the results of sifting.

Practical Application

In the hope of having something tangible upon which to work in the attempt which was made to standardize the granulation test, particular attention was paid throughout to the question of the separation of the flour particles, and to study the factors and determine the extent of their influence on granulation. This problem proved to be a very complex one and involved an enormous amount of time and work.

The information gained should prove very valuable to the miller and may also serve as a foundation for further research work. In view of the fact that initial sifting will be comparable to what would be obtained in commercial sifting, the miller will be particularly concerned with the conditions which promote production in initial sifting.

The two most important factors are temperature and relative humidity and it is equally important that these be kept constant from day to day. This would overcome difficulties which are often experienced in unheated mills through the tendency of the warm products from the rolls to sweat and the stock in such a condition undoubtedly retards sifting. The heat generated by the rolls could be utilized in heating the mill, especially in the winter.

The statement just made regarding the importance of constant temperature and relative humidity agrees with findings concerning the advantages to be gained in yield and uniformity of products from the use of manufactured weather or air-conditioning in flour mills. [Miller (1924, 1928), Henkle (1928), Smith (1928), Oppen (1929).] These investigators were not so concerned with the operation itself as with the advantages to be gained.

Shollenberger did not make any conclusions regarding the combined influence of temperature and relative humidity but claimed that "with each increase of relative humidity there was a proportionate increase in the total yield of mill products." On the other hand, he maintained that "no definite relation between air temperature and total yield of mill products was established."

The very fact that the highest results in sifting are obtained under definite conditions of temperature and relative humidity goes to prove the beneficial effect of the air conditioning system. It follows that it would have a similarly advantageous effect on grinding as well, and some mills have already realized the importance of controlling these factors. It is also evident that definite conditions of temperature and relative humidity have a contributory influence on cleaning the silks, eliminating many unfavorable physical conditions which would otherwise effect the sieves. Air, with the right temperature and right moisture content, may therefore be considered a valuable "cleaner." Further, as coarse particles have in themselves a great influence on sifting, the use of cleaners would be dispensed with in sifting coarse middlings, sizings, etc., provided the temperature and relative humidity are such as to induce the best results in sifting. However commercial cleaners would influence intensity of sifting in fine middlings and tailings.

Whilst the foregoing statements are all based on actual results obtained there are other factors upon which granulation of flour is dependent. First there is the character of the wheat itself and the degree of hardness; obviously the problem becomes very complicated where several different wheats are blended. In the second place, grinding and manipulation are individual problems which will influence sifting. It would be possible for a miller, knowing his own blends and manipulation, to use the granulation test to good advantage in controlling the uniformity of his flour.

After deliberating upon the fund of information accumulated as a result of the experimental work done, it was decided to adopt the following procedure in making future granulation tests in this laboratory until such time as some other method proves itself more suitable:

1. To treat the flour or flours so that they will have 13.00%-13.50% moisture.

2. To use the method of continuous sifting, one lot of 100 grams of flour sufficing for the whole experiment.

3. To use 20 gms. of wheat as a cleaner.

4. To perform the test under conditions of 70°F. (21.1°C.) and 70% relative humidity.

5. To have flour and wheat the same temperature as room, namely 70°F. (21.1°C.).

6. To record the percentage of flour passing through the silks as follows:

100 grams of 1st. Patent Baker's Flour

Silk No.	Time of Sifting in Minutes	Passing Through %
25 Stand.	½	30.03
" "	½ — 1½	15.16
" "	1½ — 3	5.10
" "	3 — 6	2.52
" "	6 — 10	2.12
" "	10 — 15	1.98
	Total through in 15 minutes	56.91
20 Stand.	15 — 20	11.40
16XX	20 — 25	8.21
13XX	25 — 30	20.06
	Total passing through	96.58
	Held up on 13XX	2.80
	Total Yield	99.38

Acknowledgment

Acknowledgment is made of the help given by Mrs. Elizabeth Child in preparing this article.

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A VOLUME-MEASURING APPARATUS FOR SMALL LOAVES [1]

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(Received for publication February 27, 1930)

Introduction

Since the loaf-measuring devices now on the market for use with small loaves are unsatisfactory either in operation or in price, it was decided to have one constructed locally to our own specifications. After consideration of the various types described in the literature or by manufacturers, the closed-system type of device, such as that manufactured by the Industrial Appliance Co., and modified by Geddes and Binnington (1928), was chosen as the most satisfactory. We have, however, modified their design in a manner which we believe increases the ease of construction and the accuracy of operation. The apparatus described in this paper has been used in our laboratory for the last two years with entire satisfaction.

Description of Apparatus

A drawing of the apparatus together with the detail of certain parts is given in Fig. 1.

The glass tube D (1″ bore) is calibrated in 10 cc. divisions. This was done by stoppering one end and adding successive 10 cc. portions of water from a standard pipette. This tube is enclosed in a length of nickeled piping into which it fits snugly. Slots are provided in the piping to allow the glass tubing to be seen. The tube is held in place in the pipe by means of metal collars soldered to the inside of the pipe. A steel ball, 7/16″ in diameter, is suspended in the center of the tube near one end by means of a stiff wire passing through the centre of the ball and soldered to the sides of the pipe. The frame for slide C is soldered between the pipe and the hopper A. The slide has two apertures, one 1 1/16 inches in diameter and the other 7/16 inches. The hoppers are made of stiff sheet brass and are identical except that A has fine wire mesh let into the sides to provide ventilation. The cover for B has clips for holding the loaf and raised rings around the slots so that it is held firmly in position by the thumb screws F when it is on the hopper. The whole apparatus is suspended in a frame which permits it to be inverted.

[1] Paper No. 12 of the Associate Committee on Grain Research, National Research Council of Canada.

The standard loaf or "dummy" has a volume of 550 cc. A tight brass box of slightly less than this volume was constructed. The exact volume of the box was found by water displacement. The volume of solder necessary to make up the volume to 550 cc. was measured out by water displacement. This was laid on top of the box and melted into place. The volume of the completed standard loaf was then checked and found to be 550 cc.

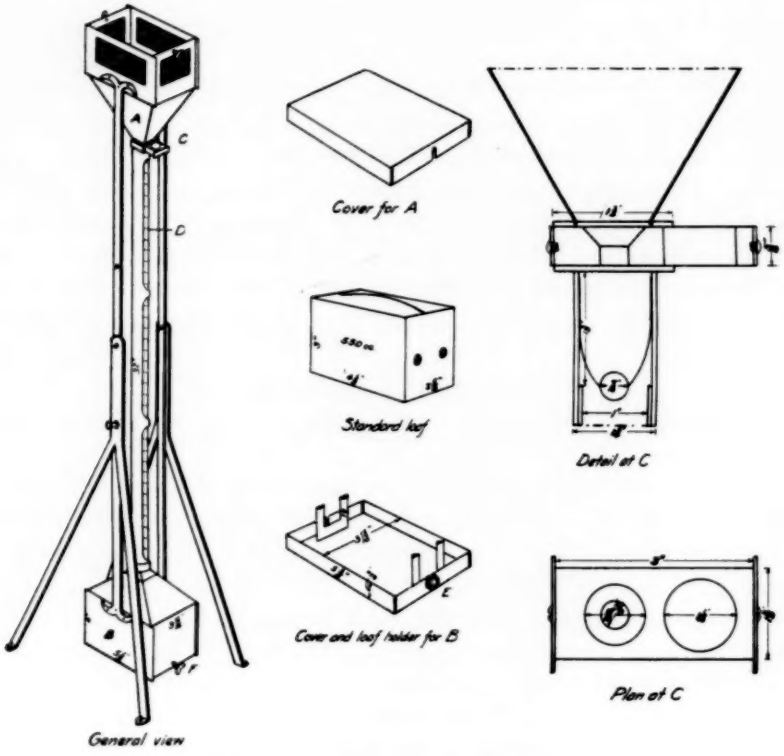

Fig. 1. Volume-measuring device, showing parts in detail.

Use of the Apparatus

The standard loaf is placed in hopper B, the apparatus is inverted, and a quantity of rape seed is poured in. The quantity is adjusted till a reading of 550 cc. is obtained on the scale. The slide C is moved until the large hole is in line with the tube. The apparatus is inverted and the seed allowed to flow into the hopper A. The "dummy" is then replaced by a loaf to be measured. The slide is adjusted so that the small opening is in line with the tube. The apparatus is inverted and the seed allowed to flow through the

small aperture and around the loaf. The volume is read directly from the scale. Adjustments for abnormally small or large loaves may be made by adding or removing definite quantities of seed. In routine practice the quantity of seed is checked by the "dummy" after every second loaf measured. This is necessary because a slight loss of seed by adhesion to the loaves is unavoidable. There are also slight changes in the size of the individual seeds owing to absorption of moisture from the loaves. No adjustment of the amount of seed is made unless the discrepancy exceeds 5 cc., but the volumes of the loaves are corrected by adding or subtracting the difference between the apparent volume of the "dummy" and 550 cc.

Accuracy of the Apparatus

It was desired to check the accuracy of the apparatus over a range of volumes. Wood blocks are not entirely satisfactory. The use of water-filled balloons as recommended by Harrel (1928) is not satisfactory since they do not even remotely approach the shape of a loaf, and the shape of the object measured may have an appreciable effect on the packing of the seed. The plan adopted made use of ordinary loaves of various volumes picked from a series of routine tests. These were allowed to dry out by standing exposed to the air in the laboratory. They were then impregnated and waterproofed with paraffin wax and their volume measured by water displacement. They were next measured in the ordinary way in the loaf-volume device. The comparative volumes obtained by the two methods are given in Table I.

TABLE I

COMPARATIVE VOLUMES OBTAINED BY WATER DISPLACEMENT AND BY THE APPARATUS

Loaf	Loaf Volume	
	Water Displacement	Apparatus
	cc.	cc.
A	279.8	281
B	392.0	390
C	467.0	468

The results by the two methods show a very satisfactory agreement.

As a further check on the accuracy of the apparatus, a record was kept of the apparent volume of the standard loaf during a series of routine bakings. These volumes are given in Table II.

TABLE II

APPARENT VOLUME OF STANDARD LOAF

Reading No.	Apparent Volume
1	551
2	552
3	552
4	552
5	552
6	552
7	550
8	550
9	549
10	551

This series shows a very consistent apparent volume which is adequate guarantee of the uniformity of packing of the seed.

Advantages of the Apparatus

The apparatus can be easily constructed and the total cost is about $35, including labor and material. Placing of the aperture at one end of the tube instead of in the middle has several advantages. It simplifies the construction and the calibration of the tube. It allows the whole length of the tube to be utilized. If the aperture is in the middle, part of the seed does not pass through it when large loaves are measured, and the packing is affected thereby.

Perhaps the most important result of this design is that the seed has a long drop and consequently very uniform packing is obtained. The placing of the steel ball in the center of the tube near one end spreads the seed as it drops: this ensures better packing and gives a flat surface to the column of seed in the tube. The efficiency of the packing may be judged by the fact that the apparatus can be severely jarred without causing a fall of the seed in the measuring tube.

The double aperture allows for quick return of the seed to the hopper A. With the normal quantity of seed in the apparatus it takes 105 seconds for the seed to flow out of hopper A and only 15 seconds for the return.

The wire mesh in the sides of the hopper A is effective in keeping the seed dry and hence of constant volume and also in removing dust.

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BOOK REVIEW

Wheat—by W. W. Swanson (14 years Professor of Economics, University of Saskatchewan) and P. C. Armstrong (Consulting Agriculturist). 320 pp. Price $2.50. The MacMillans in Canada, Toronto.

This is a book by the leading economist of Saskatchewan, dealing with the subject that is dearest to the heart of the University of Saskatchewan—wheat. It is at once a history, and a physical description, and an economic study of the wheat marketing of Canada. It is written in popular form, with numerous statistical tables and an "economic chart of prairie provinces 1890 to 1928, showing wheat, population, immigration, homestead entries and railway constructions" (p. 321).

The book is what its title indicates, the story of "wheat" (in 17 chapters), as it is seeded, grown, cultivated, threshed, handled, and finally milled or shipped to the world's markets. It discusses the wheat country and the wheat growers' problems, and has chapters on crop forecasts and cost statistics; elevators; trains and ships; grain exchange; broker and speculator; Liverpool, where world wheat prices are fixed; and on the Canadian Government in its relation to the wheat grower. One chapter covers the Building of the Co-operative System and the Wheat Pools,—which latter the authors term "the wisest move ever made by the Western farmer, and his brightest hope."

The book is both practical and analytic, as a few sentences selected here and there will indicate.

Transportation costs seem to be paid and borne by the buyer, and when navigation closes, raising rates ten cents, the wheat market at Winnipeg does not drop. A terminal elevator at Fort Churchill (on the Hudson's Bay) costing $2,000,000 is now under construction.

> "The improvement of the St. Lawrence Waterway * * * is probably of interest chiefly as a power project."
>
> "We incline to the belief that the time has come * * * to suggest that the grading and certificating of wheat for export should be divided into its two entirely distinct parts. The present system should be applied in order to obtain the grade on which the producer is to sell his grain; another and entirely new inspection should be applied to wheat when it is sold for export. The second inspection might well be applied when the wheat is placed in the ship in which it is to move overseas. Incidentally [this] might remove the danger of tampering with Canadian grading in the United States ports."
>
> "The time has probably come to consider taking the definition of grades of Canadian grain out of the hands of the national Parliament, and placing them as a responsibility on the men who produce and sell the crop."

As regards Russia, where Prof. Swanson has recently been, he thinks it is not likely to become a serious threat.

> "The very success of the [Russian] Administration in its task of economic improvement of the workers' position will defeat any hope of the creation of export surpluses of foodstuffs."

They will, if prosperous, buy and eat it themselves. The world wheat market "may be described as a potential seller's market, actually operated as a buyer's" [market]. The sellers might well dominate but actually do not.

The authors think that while Canadian wheat crops will continue to increase, there is no reason to fear that the augmenting world demand (increasing about 200 million bushels annually on the average since 1913) will not keep up with it. They think there is not enough free homestead land left, sufficiently attractive to cause another rush of wheat growers to Canada. Most of what is left now is timber. They also think that the "mechanizing" of agriculture will be an aid to present farmers and add to their comforts rather than a stimulus to large scale "company" farming, although they foresee an increase in rented farms, supervised by owners and their agents. At present nearly one-third of Canadian farms in the West are wholly or partly rented. They think the conditions of the present Canadian country home life will improve

and develop, and that more and more people will go on the land for a home and less merely to get land that will make them a speculative profit.

Curiously enough, but plausibly, they explain how low grain prices will in Canada stimulate and increase production. When prices are low and the farmers are pressed by their creditors, their only remedy is to increase the acreage and seed more wheat.

Prof. Swanson thinks Western Canada is the only country in the world where the mechanical facilities for wheat handling are sufficiently modern and advanced to make wheat growers co-operation really feasible. He does not discuss the American situation much, but more than once, in reading the book, one is brought to see how very, very much the United States have still to learn from their younger sister, Canada, in the matter of wheat in all its aspects.

—DITLEW M. FREDERIKSEN.